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Supporting Information

PhSeBr Mediated Hydroxylative Oxidative Dearomatization of Naphthols – An Open Air Facile One-Pot Synthesis of Ketols

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Contents

Experimental Methods-

All reactions were carried out under open air in oven dried glassware with magnetic stirring. THF was dried over potassium carbonate first then by metallic sodium for use. Methanol was also dried in potassium carbonate and further dried by sodium. All other reagents were purchased from, Across organics, Sigma-Aldrich, Avra Chemicals, and HIMEDIA and used without further purification. Oven dried anhydrous Na_2SO_4 was used for drying the crude reaction mixture before chromatrography.

Chromatography was performed using (100-200mesh) silica gel. Analytical TLC was performed with .25 mm coated commercial silica gel plates (E.Merck, DC-kiesel gel 60 F_{254}) and visualized with UV light and vanillin stain. ^1H and ^{13}C NMR spectra were recorded on a Bruker (400 MHz, 100 MHz respectively). Chemical shifts are reported in delta (δ) units, chemical shift relative to deuterochloroform (7.26 Or 2.28 ppm for ^1H NMR & 77.2 for ^{13}C NMR). Data for ^1H and reported as follows- Chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity recorded as follows s= singlet, d= doublet, t= triplet, q= quartet, m= multiplate, dd= doublet of a doublet. IR spectra were examined by using of a perkin Elmer spectrum-2 spectrometer using Thin film deposit on NaCl plated and frequency reported in absorption (cm^{-1}). High Resolution Mass Spectra (HRMS) were measured in a QTOF I (quadrupolehexapole-TOF) mass spectrometer with an orthogonal Z-spray-electrospray interface on Micro (YA-263) mass spectrometer.

List of Abbreviations:

THF= Tetrahydrofuran ,

EtOAc= Ethyl acetate

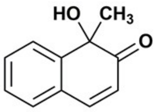
TLC= Thin layer chromatography

DCM = Dichloro methane

General experimental procedure for the hydroxylative dearomatization of napthols by PhSeBr

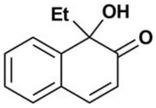
To a well stirred suspension of substituted naphthol (1mmol) & K_2CO_3 (1mmol) in dry THF at room temperature (30^0C) ,Phenyl selenyl Bromide (2mmol) was added followed by stirring for 12h (aprox.) After completion (monitored by TLC), the reaction was quenched with distilled water and followed by extracted with ethyl acetate. The organic parts were washed with brine & dried over sodium sulphate, filtered and concentrated. Then the residue was subjected to column chromatography over silica gel. Elution with EtOAc in petroleum ether afforded the desired compound.

1-hydroxy-1-methylnaphthalen-2(1H)-one 1(c)



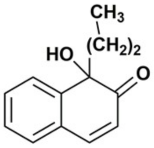
The reaction was carried out according to general experimental procedure. To a well stirred solution of 1-methyl-2-naphthol (**s_3)** (16 mg ,0.1 mmol) & K_2CO_3 (28mg,.20mmol) in dry THF (3 mL) at room temperature (30^0C) , Phenyl selenyl Bromide (48 mg, 0.204 mmol) was added followed by stirring for 6h at rt . Then the reaction mixture was quenched with distilled water and followed by extraction with ethyl acetate (5X3ml).The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 5% EtOAc afforded 1-hydroxy-1-methylnapthalen-2(1H)-one 1**(c)** 17 mg **(**97%) as a white coloured solid.R_f = 0.5(10% EtOAc in petroleum ether). The spectral data of 1(c) were identical with the reported compound.[a]

1-ethyl-1-hydroxynaphthalen-2(1H)-one 1(d)



The reaction was carried out according to the general procedure . To a well stirred solution of 1-ethyl-2-naphthol (**s_4)** (85 mg , 0.49 mmol) & K_2CO_3 (75mg, .54mmol) in dry THF (5 mL) at room temperature (30^0C) , Phenyl selenyl Bromide (220 mg, 0.93 mmol) was added followed by stirring for 8h at rt . Then the reaction quenched with distilled water and followed by extraction with ethyl acetate (5X3ml).The organic part was washed and dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 7% EtOAc afforded 1-ethyl-1-hydroxynaphthalen-2(1H)-one 1**(d) (**80 mg 87%) as a slightly coloured liquid. R_f =0.5(15% EtOAc in petroleum ether); The spectral data of 1(d) were identical with the reported compound.[a]

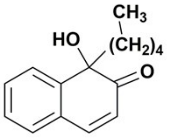
1-hydroxy-1-propylnaphthalen-2(1H)-one 1(e)



The reaction was conducted according to general experimental procedure. 1-propyl-2-naphthol (**s$_5$**) (130 mg, 0.69 mmol) ,potassium carbonate 1(144mg, 1.04mmol), Phenyl selenyl bromide (328 mg, 1.2 mmol). The reaction mixture was stirred for 8h at room temperature. Then the mixture was quenched with distilled water and followed by extraction with ethyl acetate (5X3ml). The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Using 7% EtOAc furnished the 1-hydroxy-1-propylnapthalen-2(1H)-one **1(e**) as a pale yellow liquid (127 mg, 91 %). R$_f$ =0.55 in 10% EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3450, 3246, 1680, 1450,750, 550 cm^{-1}. ^{1}H NMR (400 MHz, CDCl$_3$) 7.64(d,*J*=7.6, 1H), 7.41(m,2H), 7.31(m,2H), 6.16(d,*J*= 9.6Hz,1H), 3.78(s,1H), 1.74(m,2H), 1.20 (m,2H), δ 0.812 (t,*J*=6.8Hz, 3H) ;^{13}CNMR (100 MHz, CDCl3) 205.5, 145.82, 144.53, 130.16, 129.31, 128.86, 127.71, 126.00, 122.75, 80.13, 47.68, 17.09, 15.28,HRMS (ESI) calc'd for $C_{13}H_{14}O_2Na$ [M+Na]+ : 225.0994, Found 225.0974.

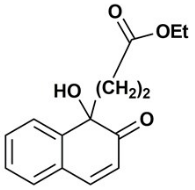
1-Hydroxy-1-pentylnaphthalen-2(1H)-one 1(f)



The reaction was carried out according to general experimental procedure. 1-pentyl-2-naphthol (**s$_6$**) (22 mg, 0.102 mmol), potassium carbonate (61mg,0.205 mmol), Phenyl selenyl bromide (48 mg, 0.204 mmol).The reaction mixture was stirred for 7h in room temperature. Then the reaction were quenched with distilled water and followed by extraction with ethyl acetate (5X3ml).The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 5% EtOAc we get our desired 1-hydroxy-1-pentylnaphthalen-2(1H)-one **1(f) (**23 mg 98 %) as a colourless liquid. R$_f$=0.49 in 10 % EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3444, 1635, 1275,750 cm^{-1}. ^{1}H NMR (400 MHz, CDCl$_3$) δ7.63(d,*J*=8.4,1H), 7.41(m,2H), 7.28(m,2H), 6.16(d, *J*=10Hz,1H), 3.76(s,1H), 1.697-1.796(m,2H),1.203-1.259(m,4H), 1.129-1.151(m,2H), 0.99(t, *J*=6.8,3H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 205.5, 145.7, 144.6, 130.1, 129.1, 128.8, 127.6, 125.9, 122.7, 80.1, 45.5, 31.6, 23.3, 22.3, 13.9;HRMS (ESI) calc'd for $C_{15}H_{18}O_2Na$ [M+Na]+ : 253.1307, Found 253.1291.

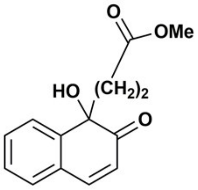
Ethyl 3-(1-hydroxy-2-oxo-1,2-dihydronaphthalen-1-yl)propanoate 1(h)



The reaction was conducted according to general procedure A .Ethyl 3-(2-hydroxynaphthalen-1-yl) propanoate (**s₈**) (40 mg, 0.163 mmol), potassium carbonate (45mg,0.327mmol), Phenyl selenyl bromide (77 mg, 0.327 mmol) . The reaction was stirred for 12 h at room temperature. Removal of the solvent furnished the red oil which was subjected to column chromatography using silica gel by elution of 10 % EtOAc in petroleum ether furnished the Ethyl 3-(1-hydroxy-2-oxo-1,2-dihydronaphthalen-1-yl) propanoate **1(h)** as a pale yellow liquid (28 mg , 67%). R_f =0.5 in 30 % EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3477, 2980,1731,1678,1070, 758 cm^{-1}.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64(d, *J*=7.6Hz 1H), 7.42(m,2H), 7.30 (m,2H), 6.17 (d, *J*= 10 Hz, 1H), 4.04(q,*J*= 7.2 Hz,2H,), 3.82(s, 1H), 2.32 (t, *J*= 7.6, 2H), 2.11(m,2H), 1.20(t,*J*= 7.2, 3H); ^{13}CNMR (100 MHz,$CDCl_3$) δ , 204.8, 172.8, 145.8, 143.5, 130.2, 129.5, 128.8, 128.07, 126.1, 122.5, 78.9, 60.5, 39.2, 28.5, 14.1;HRMS (ESI) calc'd for $C_{15}H_{16}O_4Na$ [M+Na]+ : 283.1049, Found 283.0948.

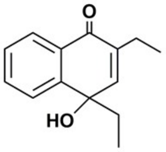
Methyl 3-(1-hydroxy-2-oxo-1,2-dihydronaphthalen-1-yl)propanoate 1(i)



The reaction was conducted according to general procedure A . To a stirred suspension of Methyl 3-(2-hydroxynaphthalen-1-yl) propanoate (**s₉**) (35 mg, 0.15 mmol) & potassium carbonate (35mg, 0.25mmol), Phenyl selenyl bromide (72 mg, 0.305 mmol) was added. The reaction was stirred for 12 h at room temperature. Then the reaction mixture was filtered by normal filter paper & the filtrate was quenched with distilled water. Extracted by (3X3ml EtOAc), Removal of the solvent furnished the red oil which was subjected to column chromatography using silica gel by elution of 10 % EtOAc in petroleum ether furnished the Methyl 3-(1-hydroxy-2-oxo-1,2-dihydronaphthalen-1-yl) propanoate **1(i)** as a pale yellow liquid (27 mg , 73%). R_f =0.3 in 20 % EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3450, 2980,1735,1680,1065, 750 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65(d, *J*=7.2Hz , 1H), 7.42-7.46(m,2H), 7.31-7.37 (m,2H), 6.20(d, *J* = 10 Hz, 1H), 3.83(bs, 1H), 3.62 (s, 3H), 2.36(t, *J* = 7.6 Hz, 2H), 2.05-2.19(m, 2H) ^{13}CNMR (100 MHz,$CDCl_3$) δ , 204.7, 173.1, 145.8, 143.3, 130.1, 129.5, 128.7, 128.0, 126.0, 122.3, 78.8, 51.6, 39.1, 28.1 ; HRMS (ESI) calc'd for $C_{14}H_{14}O_4$ [M]+ : 246.0892, Found 246.0880.

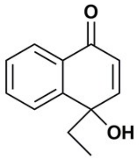
2, 4-diethyl -4-hydroxynaphthalen-1(4H)-one 1(k)



To a well stirred solution of 2,4-diethylnaphthalen-1-ol (**s$_{13}$**) (25 mg, 0.125 mmol) , K_2CO_3 (20mg,0.144 mmol) in dry THF (3 mL) at room temperature (35^{0}C) , Phenyl selenyl Bromide (60mg mg, 0.254 mmol) was added followed by stirring for 8h at rt . Then the reaction were quenched with distilled water and followed by extraction with ethyl acetate. The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 7% EtOAc afforded 2,4-diethyl -4-hydroxynaphthalen-1(4H)-one **1(k)** as a colourless liquid, (21 mg, 77 %). R_f=0.45 in 15 % EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3483, 2929, 1669,1614,1456,1374 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68(dd,*J* =1.2, 7.6Hz, 1H); 7.56 (dd,*J* =0.8, 8Hz,1H), 7.44(ddd,*J* =1.2, 7.6 Hz), 7.35(ddd, *J* =1.2, 7.6Hz, 1H), 6.12(s,1H), 3.82(s, 1H), 2.77(m,2H), 1.82(m,2H), 1,32(t,*J* =7.6 Hz, 3H), 0.83 (t, *J* =7.6 Hz, 3H); ^{13}CNMR (100 MHz, CDCl3) δ 204.9, 158.9, 144.3, 129.8, 129.5(C^3H), 127.4, 125.9, 125.0, 119.8, 79.8, 38.9, 25.9, 12.5, 8.2 . HRMS (ESI) calc'd for $C_{14}H_{16}O_2Na$ [M+Na]+ : 239.1150 Found 239.0435;

4-ethyl-4-hydroxylnaphthalen-1(4H)-one 1(l)

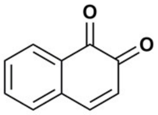


To a well stirred solution of 4-ethylnaphthalen-1-ol (**s$_{14}$**) (20 mg, 0.116 mmol) , K_2CO_3 (16mg,0.116 mmol) in dry THF (3 mL) at room temperature (35^{0}C) , Phenyl selenyl Bromide (55mg,0.232mmol) was added followed by stirring for 8h at rt . Then the reaction were quenched with distilled water and followed by extraction with ethyl acetate. The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel.

Elution with 5% EtOAc afforded 4-ethyl-4-hydroxylnaphthalen-1(4H)-one) 1(l)as a semi solid material (14 mg, 65 %) **.** R_f=0.50 in 10 % EtOAc in petroleum ether.

IR (Neat Film, NaCl) 3500, 3060, 2925, 1674,1610,1456,1370 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 8.11(d, *J* = 7.6 Hz, 1H), 7.74(d, *J* = 8Hz, 1H), 7.66(t, *J* = 8.4Hz, 1H), 7.47(t, *J* = 7.6Hz,1H), 6.94 (d, *J* = 10.4 Hz, 1H), 6.45,(d, *J* = 10.4, 1H), 2.05(m, 2H), 0.624(t, *J*=7.6 Hz,3H); ^{13}CNMR (100 MHz, CDCl3) δ 205.9, 157.0, 151.5, 133.3, 128.8, 128.1, 126.3, 126.0, 121.0, 76.0, 31.9, 8.3; HRMS (ESI) calc'd for $C_{12}H_{12}O_2Na$ [M+Na]+ : 211.0837 Found 211.0920.

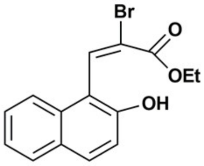
Naphthalene-1,2-dione 1(j)



The reactions were carried out according to general experimental procedure.

To a well stirred solution of 1-halogenated-2-naphthol (X=Cl,Br, I) (1mmol) & K_2CO_3 (1mmol) , in dry THF at room temperature (30^0C) , Phenyl selenyl Bromide (2mmol) was added followed by stirring at rt about 18h . Then the reaction were quenched with distilled water and followed by extraction with ethyl acetate. The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 10% EtOAc in petroleum ether afforded naphthalene-1,2-dione 1(k) in good yield. In case of Iodo-naphthol we get our desired dione in crude reaction mixture along with the selenated compound. The spectral data were identical with the reported compound.[b] R_f=0.3 in 15 % EtOAc in petroleum ether.

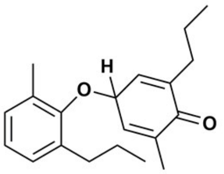
(E)-ethyl t-bromo-3-(2hydroxynaphthalen -1-yl) acrylate 1(m)



The reaction was conducted according to general procedure. (E) -ethyl 3-(2-hydroxynaphthalen-1-yl) acrylate (**s_{15}**) (75 mg, 0.309 mmol), potassium carbonate (86mg, 0.61mmol) & phenyl selenyl bromide (108 mg, 0.45 mmol). The reaction mixture was stirred for 12 h at room temperature. Then solvent were removed from the reaction mixture and the mixture separated by TLC. After purification we get (38 mg, 40%) (E)-ethyl t-bromo-3-(2hydroxynaphthalen -1-yl) acrylate **1(m)** along with some inseparable mixture. R_f= 0.51 in 20% EtOAc in petroleum ether.

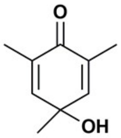
IR (Neat Film, NaCl) 2950, 1678, 823,750 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17(d,*J*=8Hz,1H), 8.03(s,1H), 7.97(d ,*J*=8Hz,1H), 7.88(d, *J*=8.8Hz,1H), 7.72(d, *J*=9.2 Hz, 1H), 7.66(t, *J*=7.2 Hz), 7.57(ddd,*J*=0.4,7.2Hz, 1H), 4.5 (q, *J*=7.2 Hz,2H), 1.48 (t, *J*=6.8 Hz,3H); ^{13}CNMR (100 MHz,$CDCl_3$) δ 160.9, 155.4, 146.4, 131.9, 130.5, 130.4, 129.4, 128.6, 126.8, 124.7, 124.1, 114.2, 62.9, 15.8 ; HRMS (ESI) calc'd for $C_{15}H_{15}O_3$ [M]+ : 320.0048, Found 320.0040

2-methyl-4-(2-methyl-6-propylphenoxy)-6-propylcyclohexa-2,5-dienone (2a)



To a well stirred solution of 2-methyl-6-propylphenol(**s_{16}**) (75 mg, 0.5 mmol) , K_2CO_3 (70mg,0.5 mmol) in dry THF (5 mL) at room temperature under nitrogen atmosphere, Phenyl selenyl Bromide (118mg, 0.5 mmol) was added followed by stirring for 12h at rt . Then the reaction were quenched with distilled water and followed by extraction with ethyl acetate (3X3) ml. The organic part was dried over sodium sulphate, filtered and concentrated. Then the residue subjected to column chromatography over silica gel. Elution with 2% EtOAc afforded 2-methyl-4-(2-methyl-6-propylphenoxy)-6-propylcyclohexa-2,5-dienone **2(a**) 55 % as a yellow colour liquid, . R_f= 0.6 in 15 % EtOAc in petroleum ether. IR (Neat Film, NaCl) 2956, 1688, 923,755 cm^{-1} .^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18-7.33(m,5H), 6.52-6.58(m,1H), 6.51-6.52(m,1H), 4.80(s, 1H), 2.56(t, *J*=7.6 Hz, 2H), 2.40-2.44(m,2H), 2.24(s, 3H), 2.07(s.3H), 1.61-1.67(m,2H), 1.53-1.58(m,2H), 0.97(m,6H); ^{13}CNMR (100 MHz,$CDCl_3$) δ 187.9, 152.2, 149.3, 145.9, 135.6, 135.0, 133.6, 133.0, 132.4, 130.4, 129.0, 128.9, 126.1, 124.4, 118.7, 77.2, 31.9, 31.1, 22.6, 21.0, 16.0, 15.8, 13.9, 13.7; HRMS (ESI) calc'd for $C_{20}H_{26}O_2$ [M]+ : 298.1933, Found 298.1928.

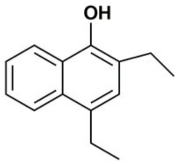
4-Hydroxy-2,4,6-trimethylcyclohexa-2,5-dieneone (2b)



The reaction was conducted according to general procedure. In a well stirred solution of 2,4,6-trimethyl phenol (**s_{17}**) (136 mg, 1 mmol) & potassium carbonate (138mg, 1mmol) in 5ml dry THF phenyl selenyl bromide was added (236 mg, 1 mmol). Then the reaction mixture was stirred for 10 h at room temperature under nitrogen atmposphere. After that reaction was quenched with distilled water & extracted by (3X3) ml ethyl acetate. Organic layer was separated and dried over oven dried sodium sulphate, Concentrated in reduced pressure and subjected to column chromatography over silica gel. Elution with 4% ethyl acetate in petroleum ether afforded the 4-Hydroxy-2,4,6-trimethylcyclohexa-2,5-dieneone **2(b)** (50mg, (85 mg, 56%) as a yellow liquid. R_f= 0.31 in 20% EtOAc in petroleum ether. IR (Neat Film, NaCl) 3350, 1682, 970,750 cm^{-1} .^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63(s, 2H), 1.87(s, 6H), 1.42(s, 3H); ^{13}CNMR (100 MHz, $CDCl_3$) δ 186.7, 147.3, 133.3, 67.1, 26.9, 15.7; HRMS (ESI) calc'd for $C_9H_{11}O_2$ Na [M + Na]+ : 175.0837, Found 175.0821.

Preparation of starting Materials-

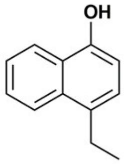
Synthesis of 2,4-diethylnaphthalen-1-ol (s_{13})



In a two-necked flask with a dropping funnel & reflux condenser, placed (4g, 0.03mol) of $AlCl_3$ in 30 ml CS_2, attached a gas absorption trap to the top of the condenser. Stir the suspension and added naphthalen-1-yl acetate (2g, 0.0107 mol) drop wise, when all the ester are added, the reaction mixture are gently reflux about 1h , then CS_2 distilled out from the reaction mixture, and then the crude materials heated in a oil bath at 140^0C about 2h.The reaction mixture allow to cool and then decompose the aluminium chloride complex by 1:1 HCl and then water, and workup by Ethyl acetate, organic part separated and dried over sodium sulphate then the organic part concentrated and this crude mixture subjected to column chromatography to afforded the two major compound. 2,4-Diacetyl 1-naphthol (0.6g,26 %) & 2-acetyl-1-naphthol (1g,54%).

To a solution 2, 4-Diacetyl 1-naphthol (100mg, 0.43 mmol) & catalytic amount of Perchloric acid in MeOH (5ml) was added 10% Pd/C (50 mg) . After being stirred under H_2 (70 psi) in high pressure reactor at room temperature for 24 h, then the reaction mixture was subjected to column chromatography over silica gel to give 2,4-diethylnaphthalen-1-ol **(s_{13})** (50mg, 62%) IR (Neat Film, NaCl) , 2890,1450, 1400,1020 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) 8.19(dd, *J* =3.2Hz, 6.8Hz 1H), 7.99(dd, *J*= 2, 10.4Hz, 1H), 7.56(t, *J*=7.6 Hz, 1H), 7.49(t,*J=7.4Hz*,1H), 7.09(s,1H), 5.07(s,1H), 3.04(q, *J*=7.6Hz 4H), 1.33-1.41 (m,6H); ^{13}CNMR (100 MHz, CDCl3) δ 161.1, 146.2, 135.9, 130.2, 129.9, 126.4, 125.2, 125.1, 124.8, 122.1, 25.5, 23.1, 14.9, 14.7 HRMS (ESI) calc'd for $C_{14}H_{16}O_2$Na [M+ Na]+ : 223.1099 Found 223.1092.

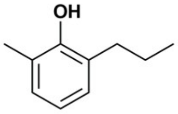
Synthesis of 4-ethylnaphthalen-1-ol (s_{14})



In a two-necked flask with a dropping funnel & reflux condenser, placed (3g, 0.023mol) of $AlCl_3$ in 30 ml CS_2, attached a gas absorption trap to the top of the condenser. Stir the suspension and added naphthalen-1-yl acetate (2g, 0.0107 mol) drop wise, when all the ester are added, the reaction mixture are gently reflux about 1h , then CS_2 distilled out from the reaction mixture. Then the reaction mixture decompose by 1:1 HCl and then water, and workup by Ethyl acetate, organic part separated and dried over sodium sulphate then the organic part concentrated and this crude mixture subjected to column chromatography to afforded the two major compound. 2,4-Diacetyl 1-naphthol (0.7g,31 %) & 4-acetyl-1-naphthol.

To a solution 4-acetyl-1-naphthol (200mg, 0.43 mmol) & catalytic amount of Perchloric acid in MeOH (5ml) was added 10% Pd/C (60 mg) . After being stirred under H_2 (70 psi) in high pressure reactor at room temperature for 24h, then the reaction mixture was subjected to column cromagatography over silica gel afforded filtered by silica filtering column to give crude 4-ethylnaphthalen-1-ol **(s_{14})**. IR (Neat Film, NaCl) 3478, 2950,1614,1425,1300 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30(d, *J*= 8Hz, 1H), 8.01(d, *J*=8Hz,1H), 7.47-7.57(m,2H), 7.16(d,*J*=7.6Hz,1H), 6.76(d, *J*=7.6Hz,1H), 3.04(q, *J* =7.6Hz, 2H), 1.36 (t, *J*=7.6 Hz, 3H); ^{13}CNMR (100 MHz, CDCl3) δ 150.8, 132.7, 131.8, 126.0, 125.1, 124.6, 124.4, 123.6, 122.6, 107.8, 25.4, 15.2; HRMS (ESI) calc'd for $C_{12}H_{12}ONa$ [M+ Na]+ : 195.2128 Found 195.2105.

Spectral data of 2-methyl-6-propyl phenol (s_{16})



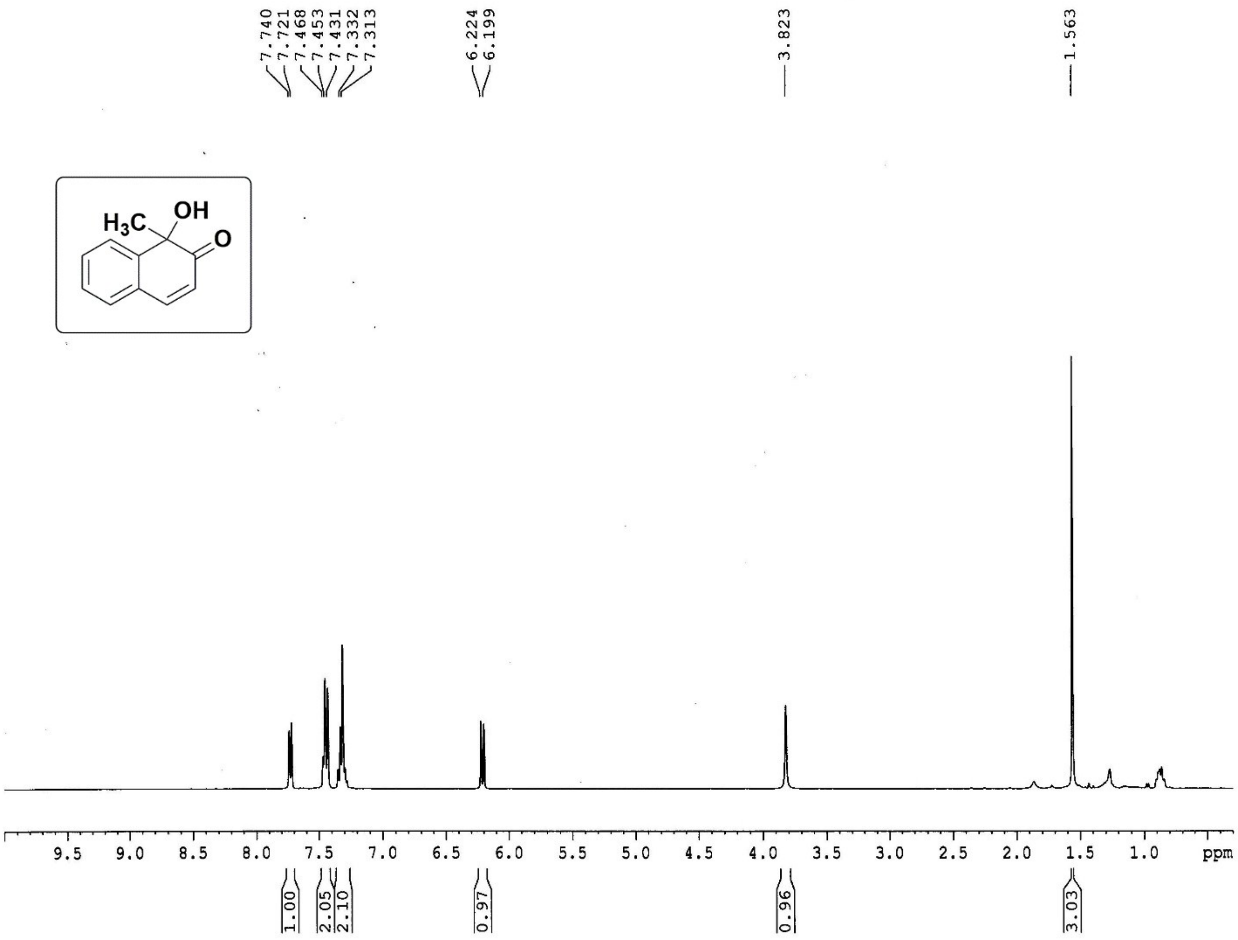
IR (Neat Film, NaCl) 3470, 2955,1300 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.0(d, *J*=7.6 Hz, 2H), 6.81 (t, *J* =7.2 Hz, 1H), 4.71 (bs, 1H), 2.60 (t, *J* = 7.6 Hz, 2H), 2.28(s, 3H), 1.63-1.72 (m,2H), 1.01(t, *J* =7.2 Hz, 3H), ^{13}CNMR (100 MHz, CDCl3) δ 151.7, 128.4, 127.8, 127.6, 123.0, 120.1, 32.1, 22.8, 15.9, 14.0; HRMS (ESI) calc'd for $C_{10}H_{14}O$ [M]+ : 150.1045 Found 150.1041.

References-

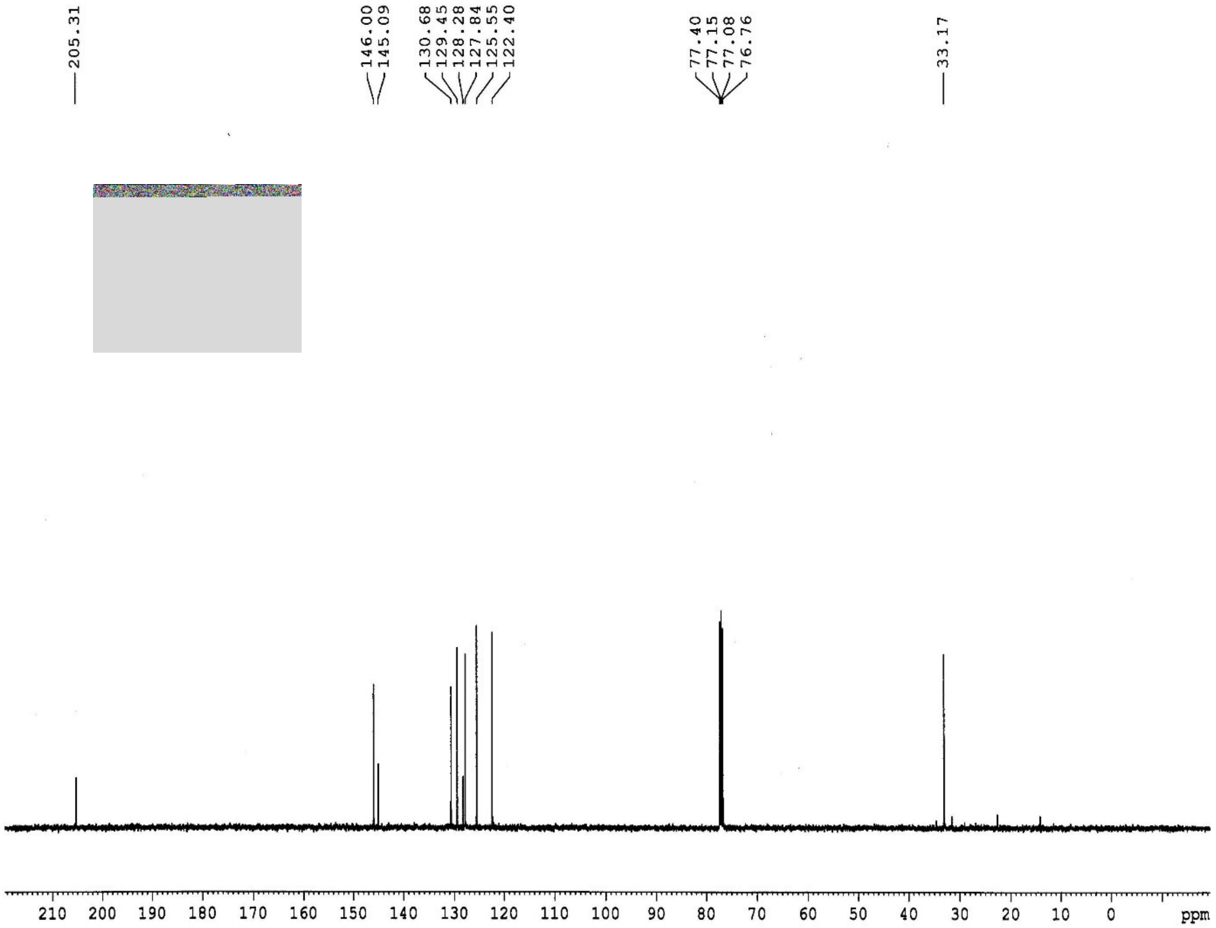
(a) K. Krohn, K. Briiggmann, D. Doring, P. J. Jones, *Chem. Ber*. 1992, ***125***, 2439;

(b) J. Ren, L. Lu, J. Xu, T. Yu, B. Zeng, *Synthesis* , 2015(june); (b) M. Uyanik , T. Mutsuga , K. Ishihara, *Molecules*. 2012, ***17***, 8604; (c) A. Wu ; Y. Duan ; D. Xu ; T.M. Penning; R.G. Harvey, *Tetrahedron* 2010, ***66***, 2111

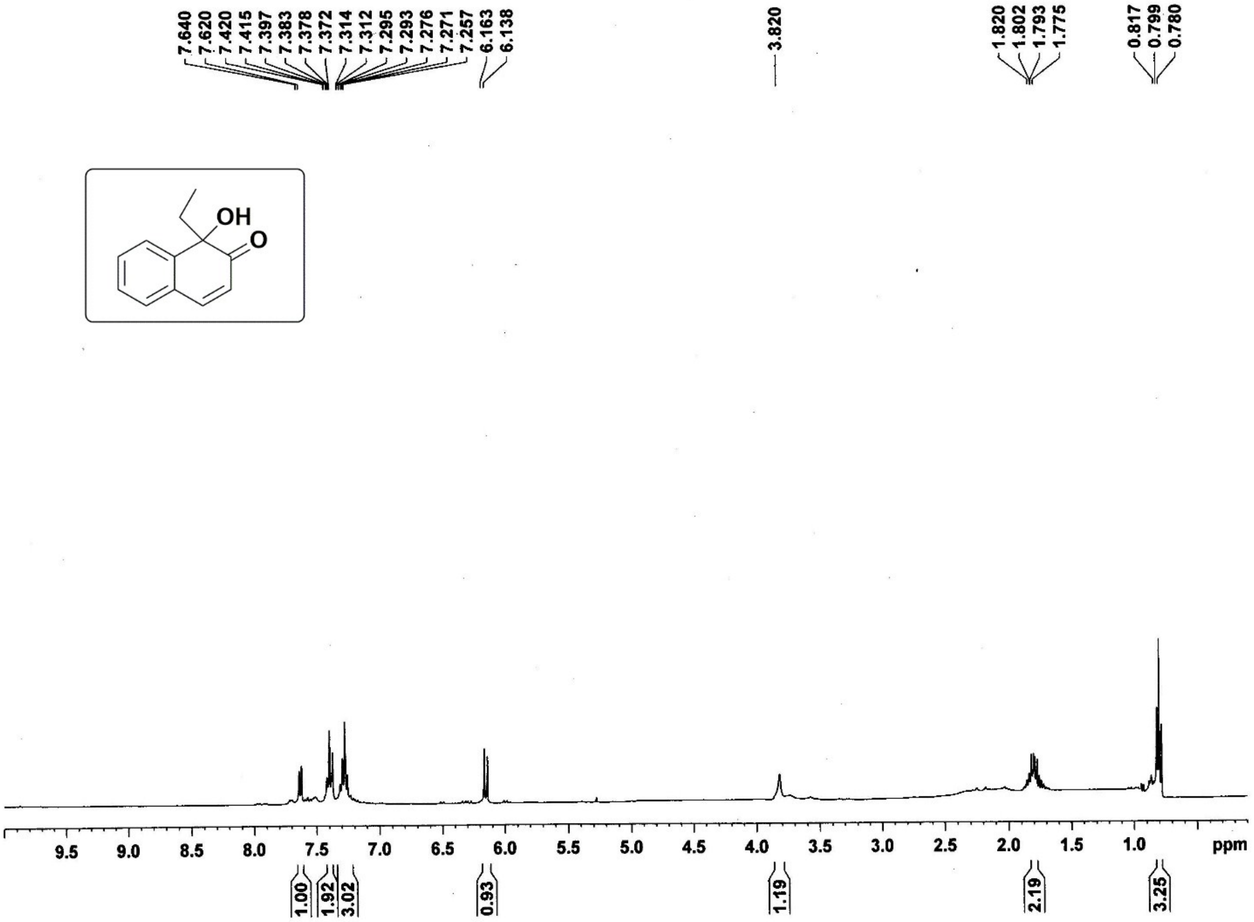
Experimental Spectra (^{1}H, ^{13}C)



^{1}H NMR (400 MHz, CDCl$_3$) of compound 1(c)

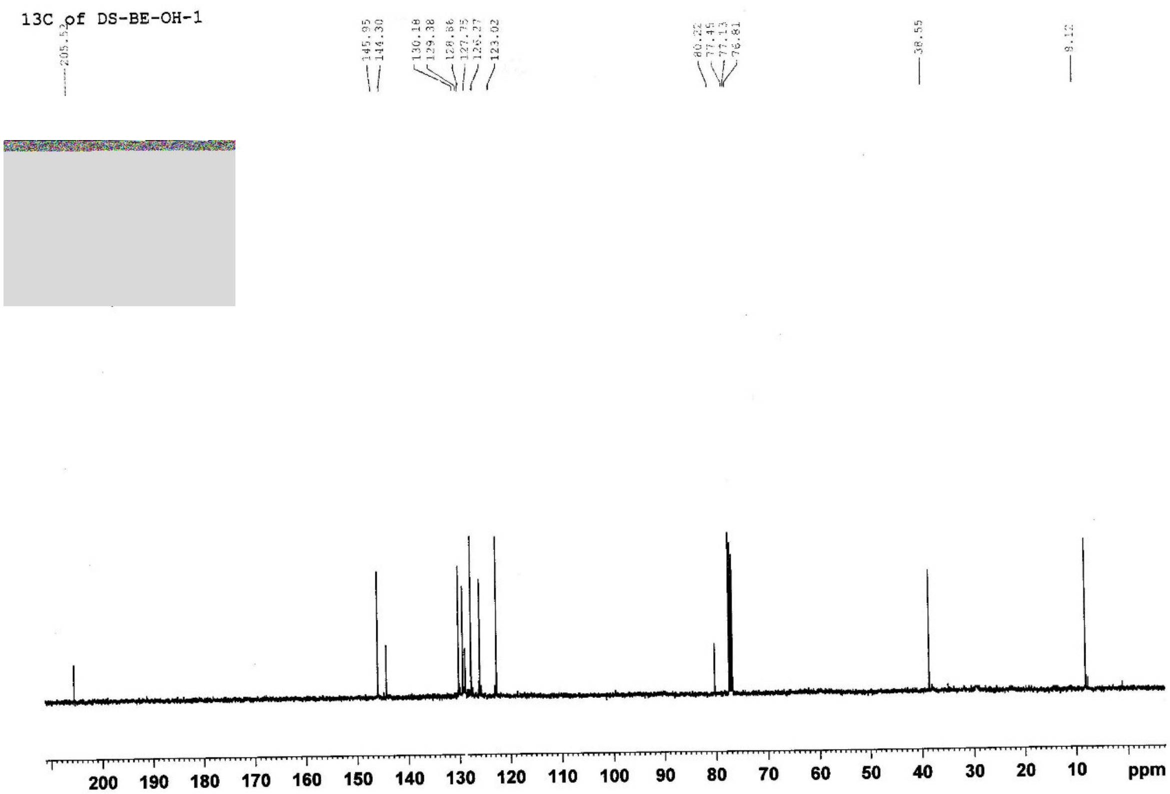


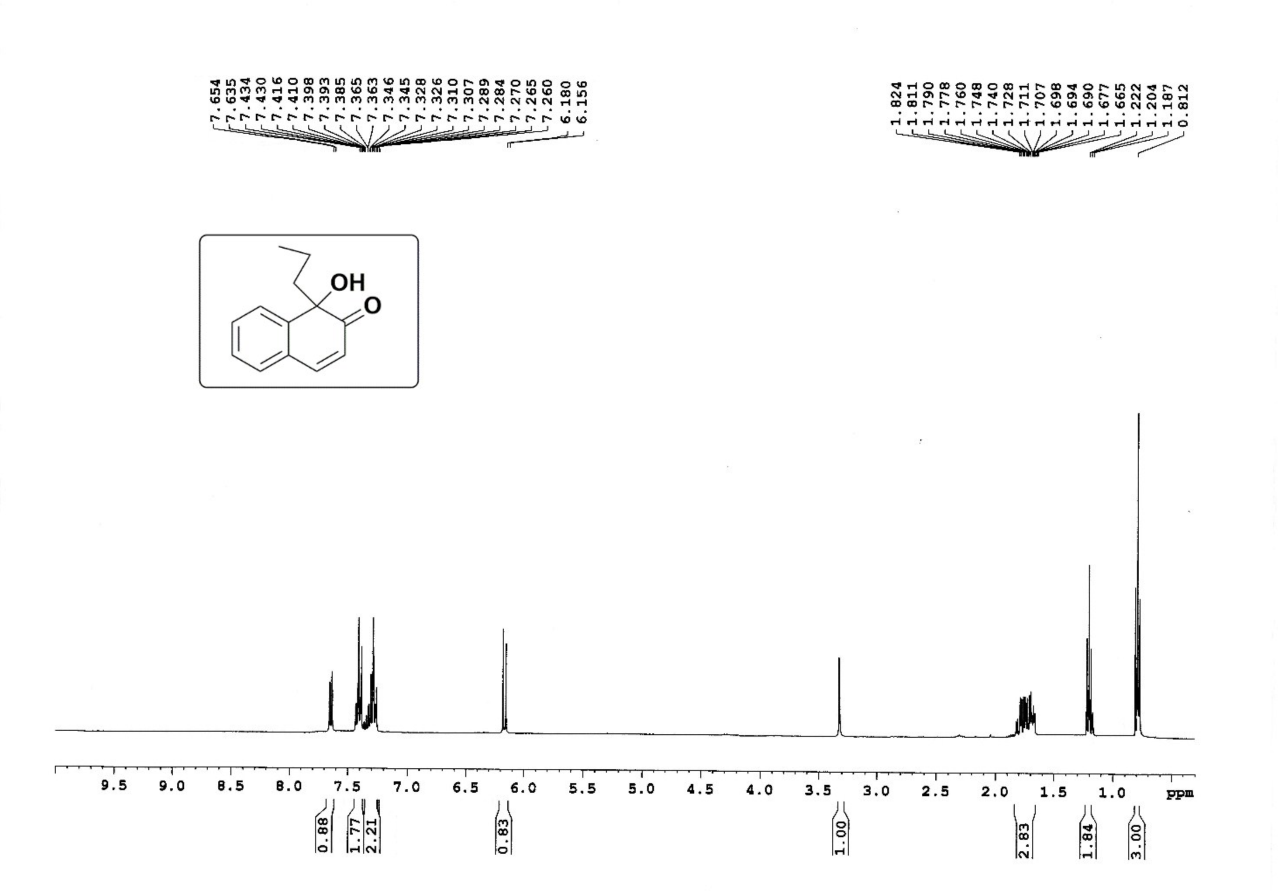
^{13}C NMR (100 MHz,CDCl$_3$) of compound 1(c)



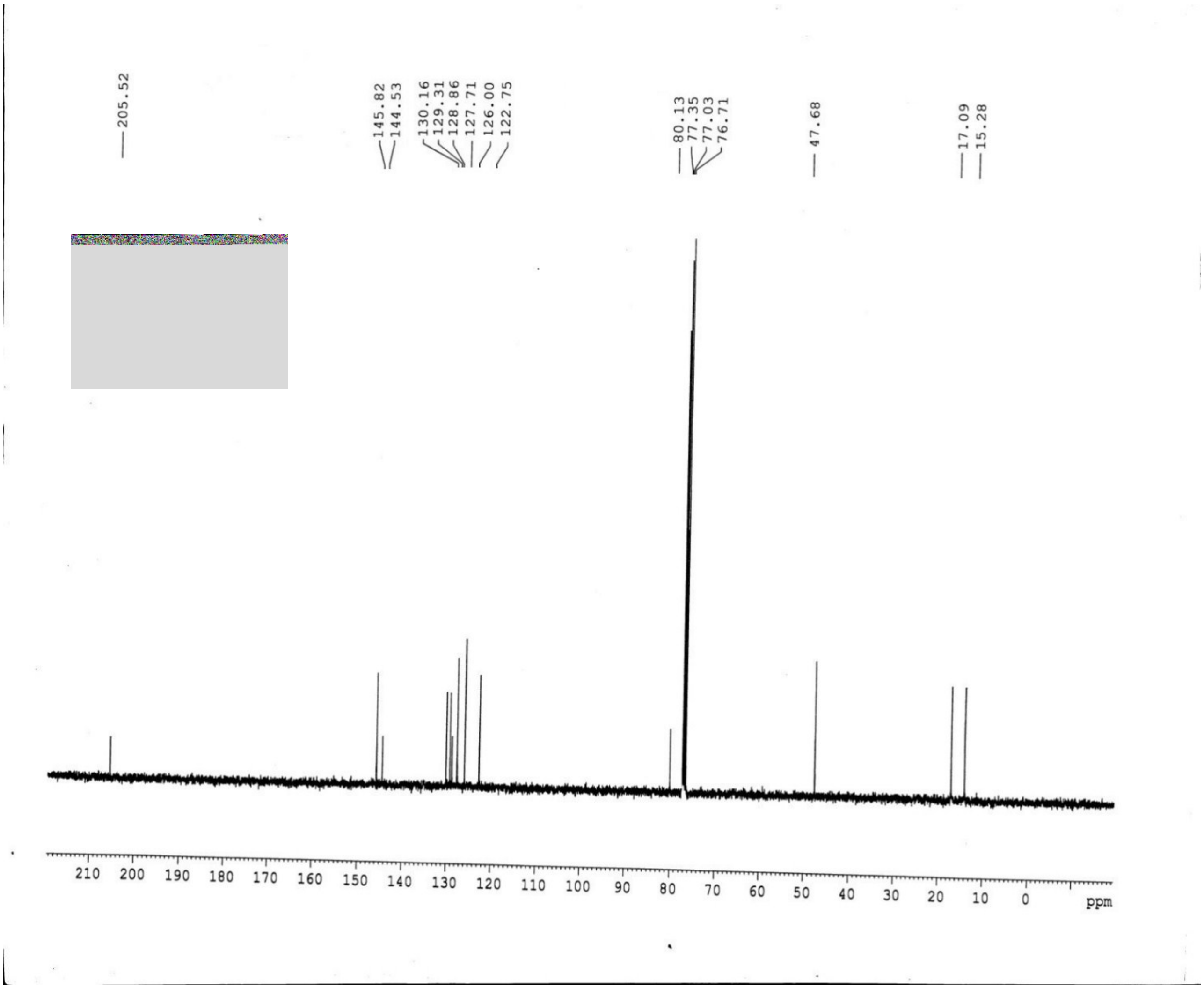
^{1}H NMR (400 MHz, $CDCl_3$) of compound 1(d)

13C of DS-BE-OH-1

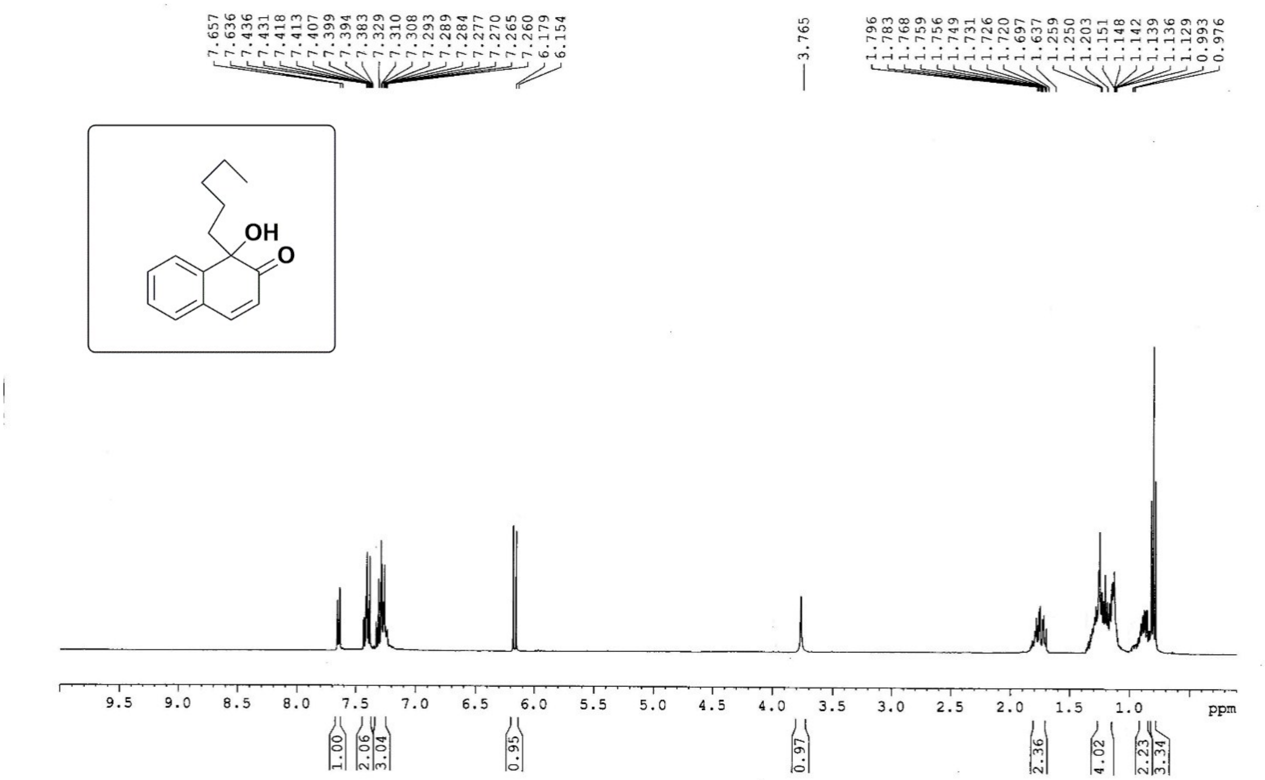




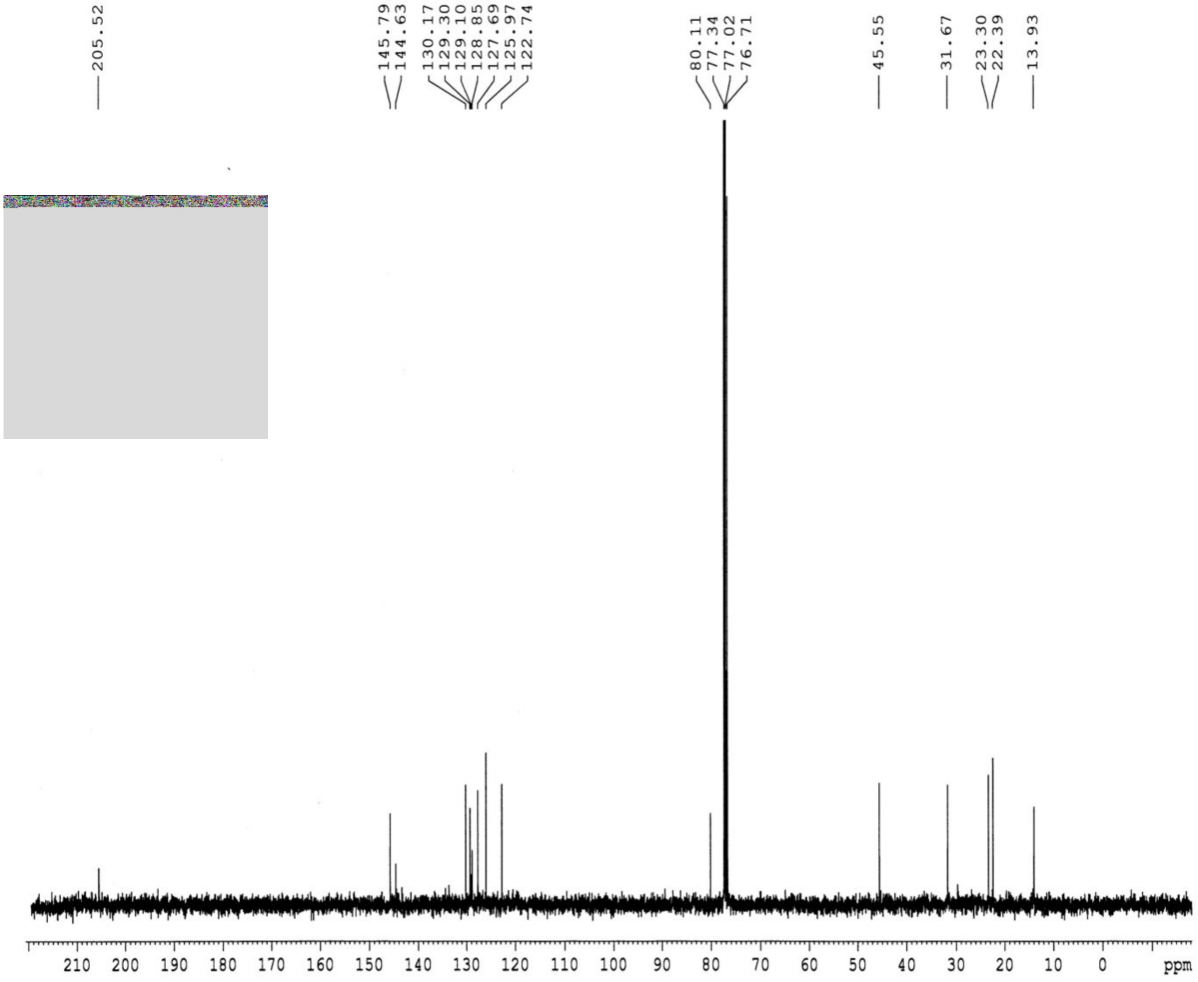
^{1}H NMR (400 MHz, CDCl$_3$) of compound 1(e)



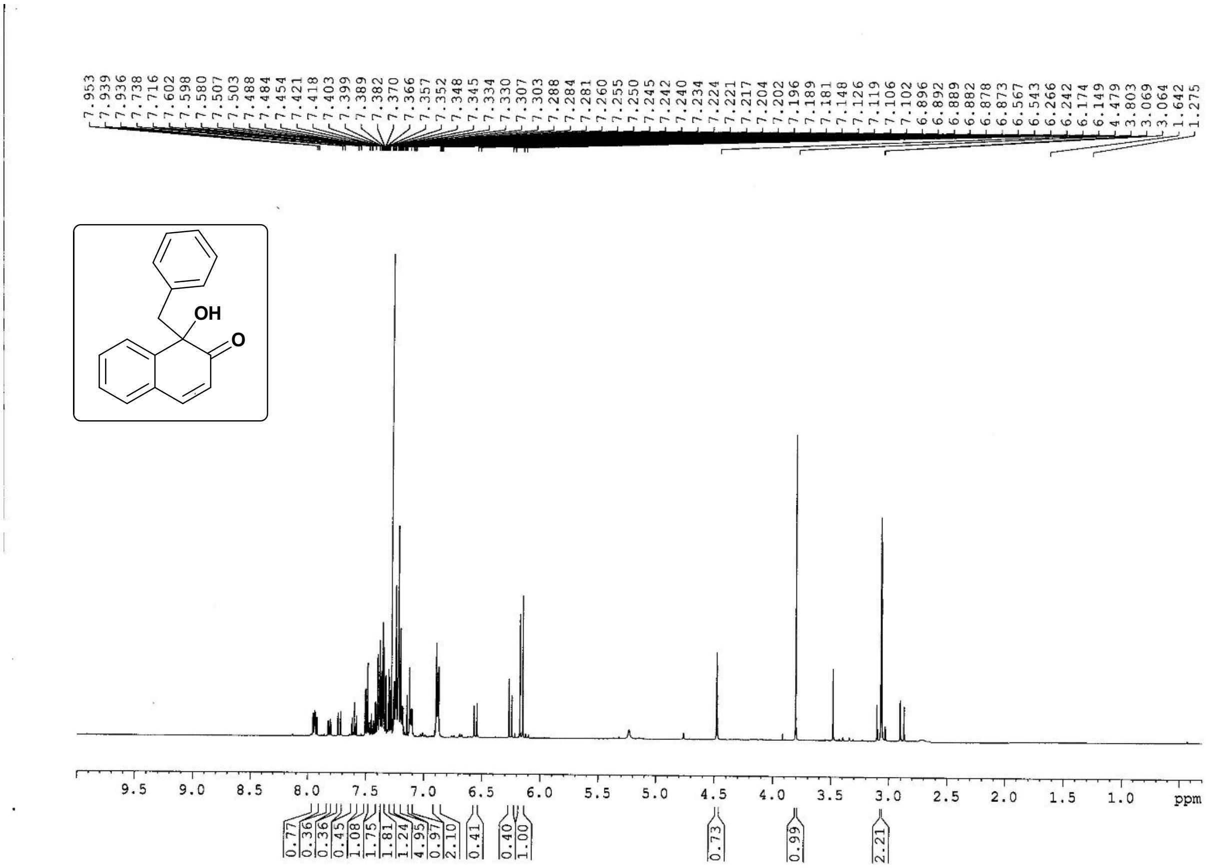
^{13}C NMR (100 MHz,CDCl$_3$) of compound 1(e)



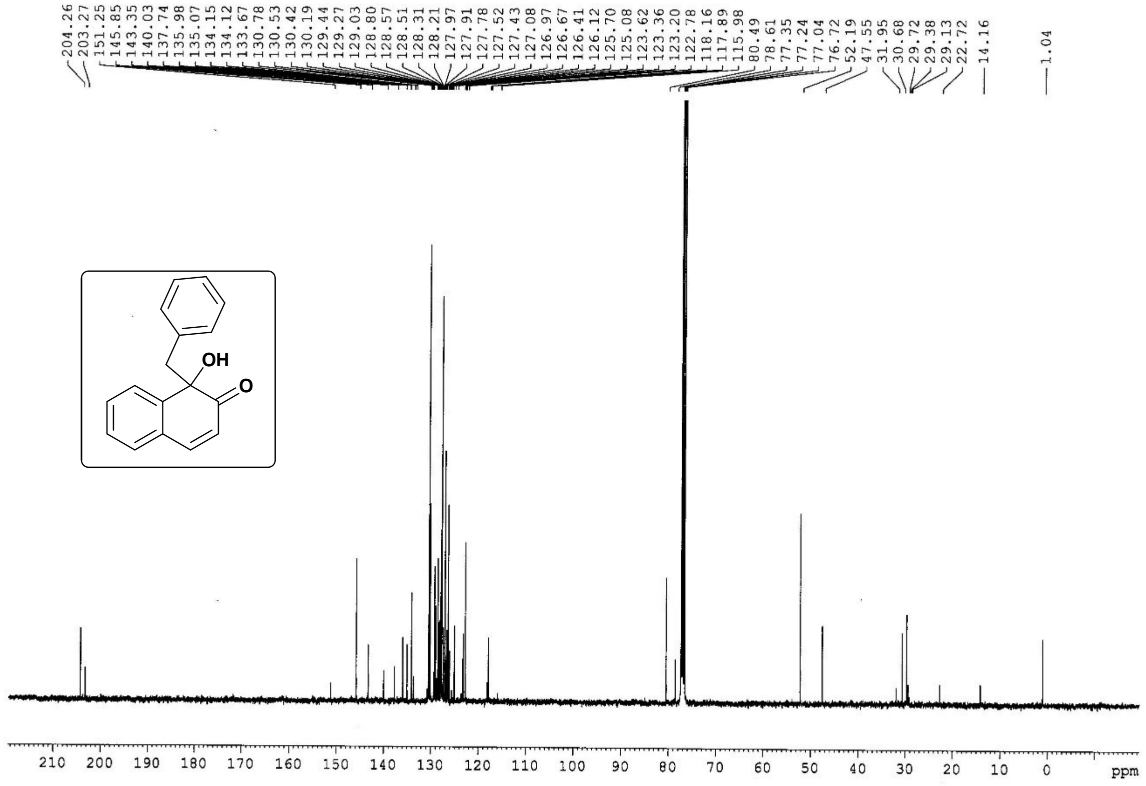
^{1}H NMR (400 MHz, CDCl$_3$) of compound 1(f)



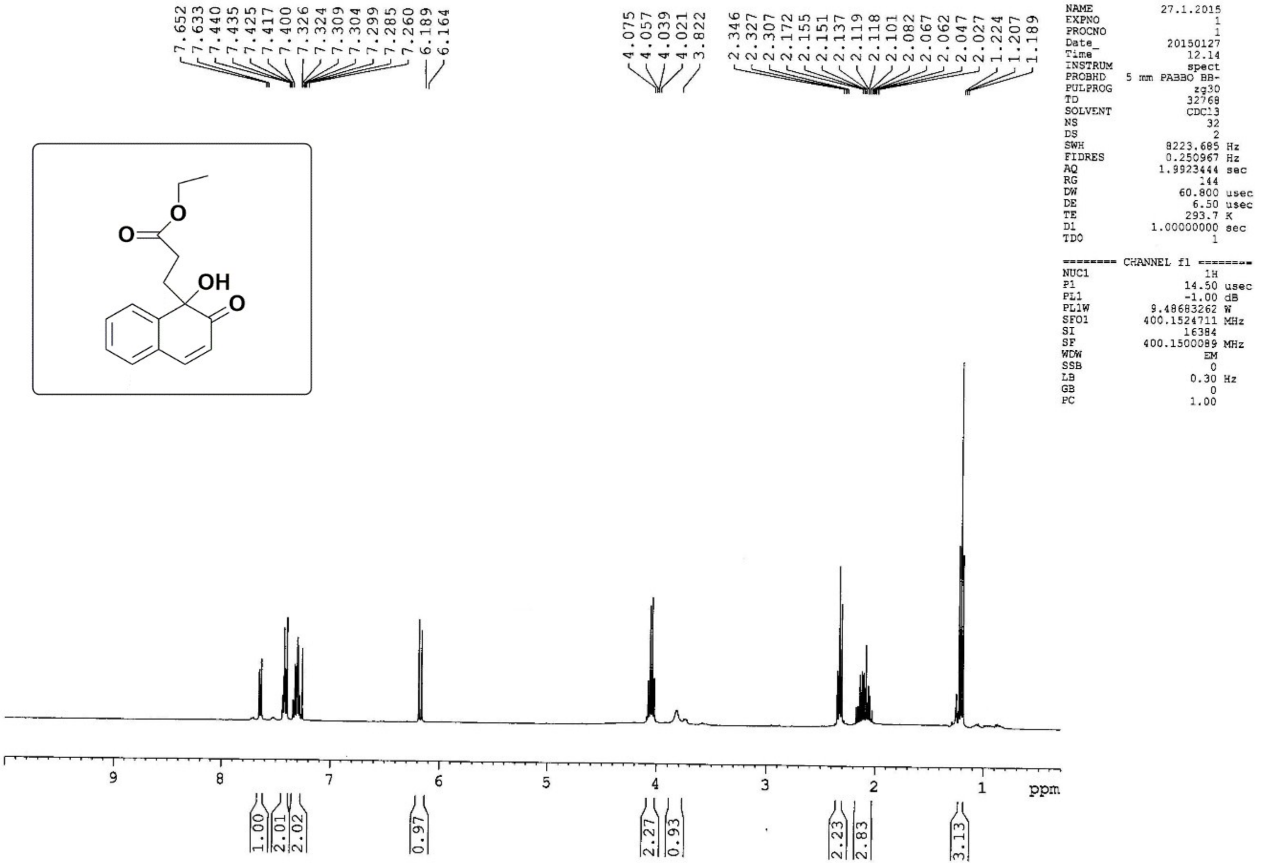
^{13}C NMR (100 MHz,$CDCl_3$) of compound 1(f)



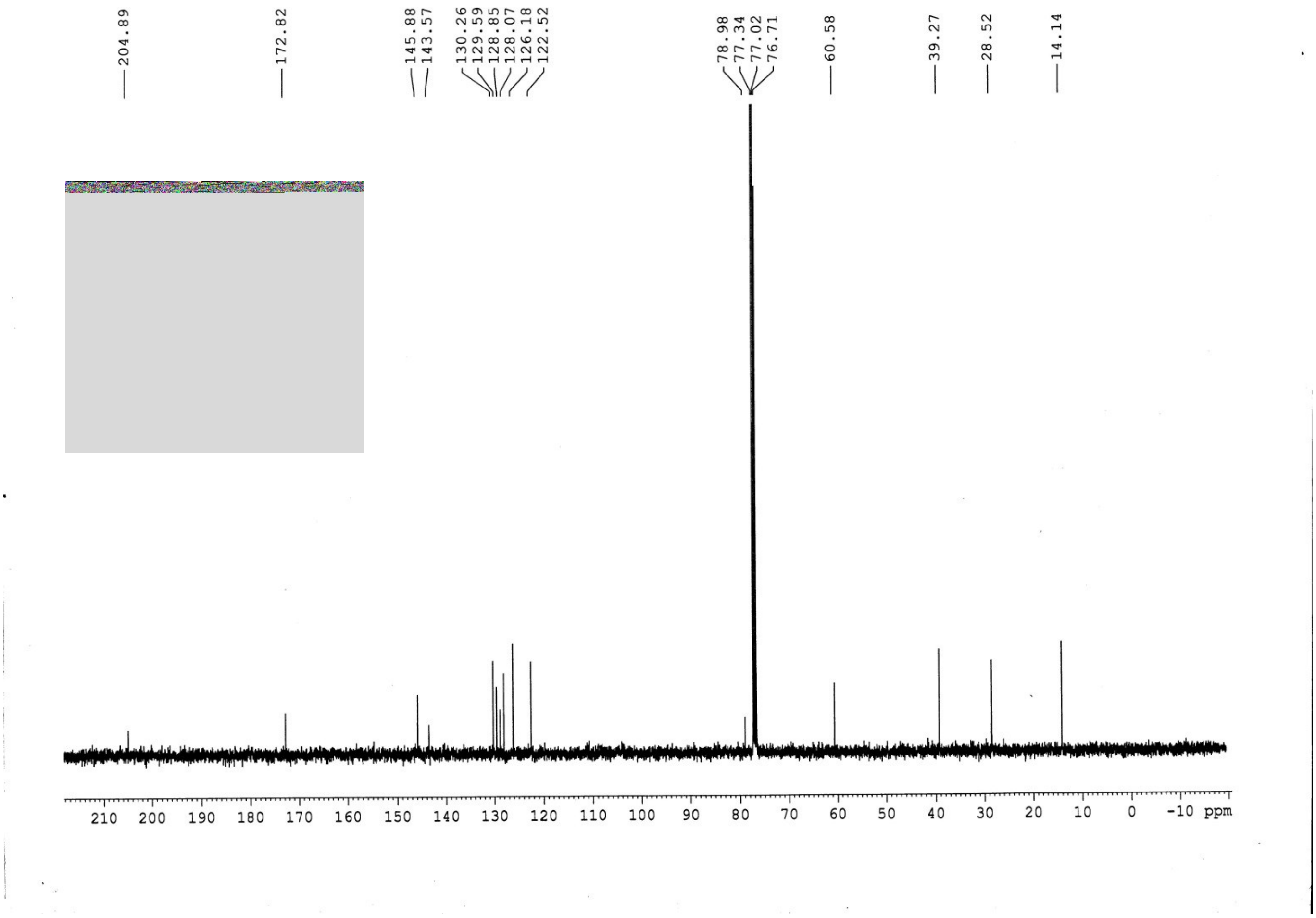
^{1}H NMR (400 MHz, CDCl$_3$) of compound 1(g)



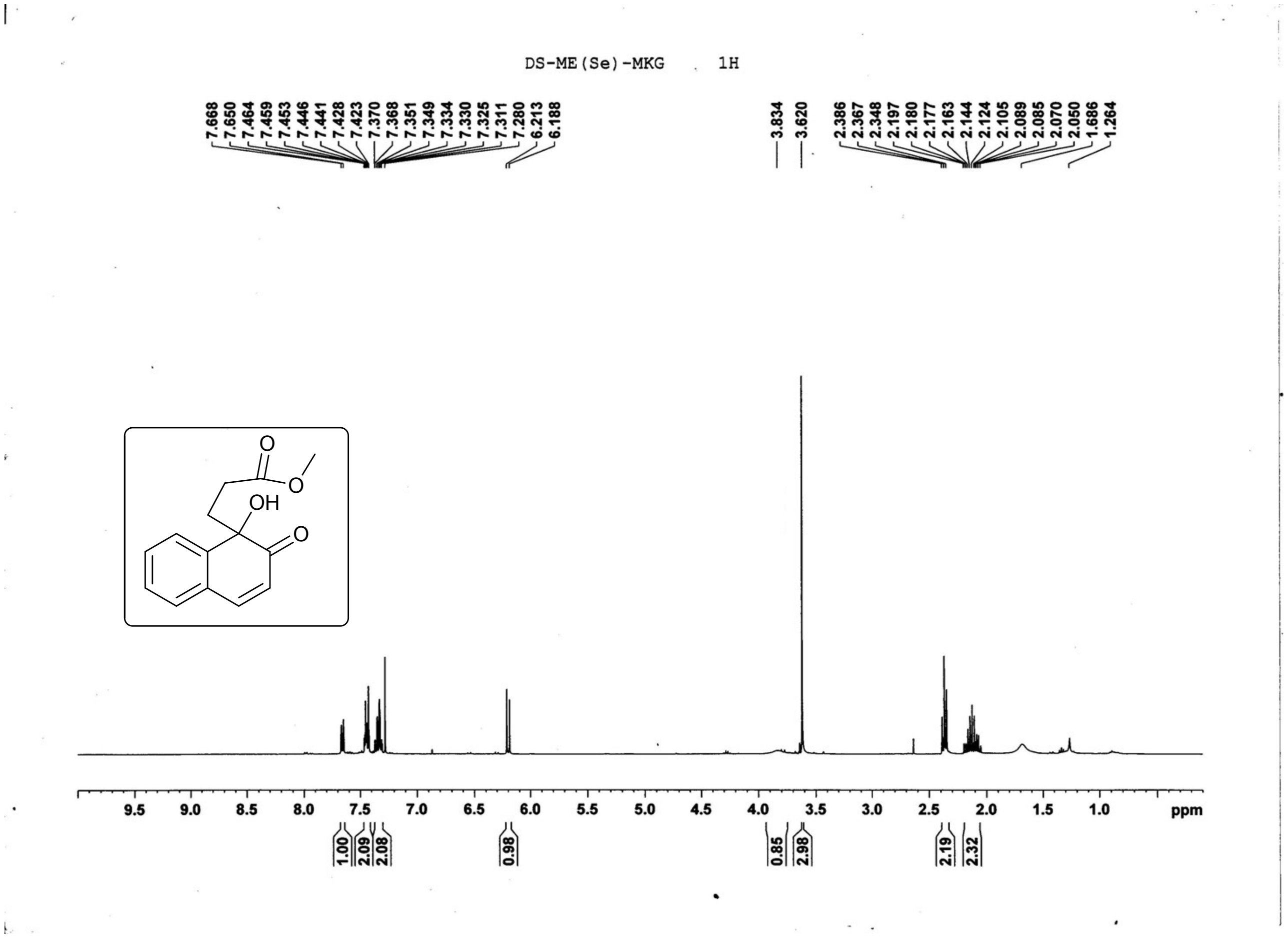
^{13}**C NMR (100 MHz, CDCl$_3$) of compound 1(g)**



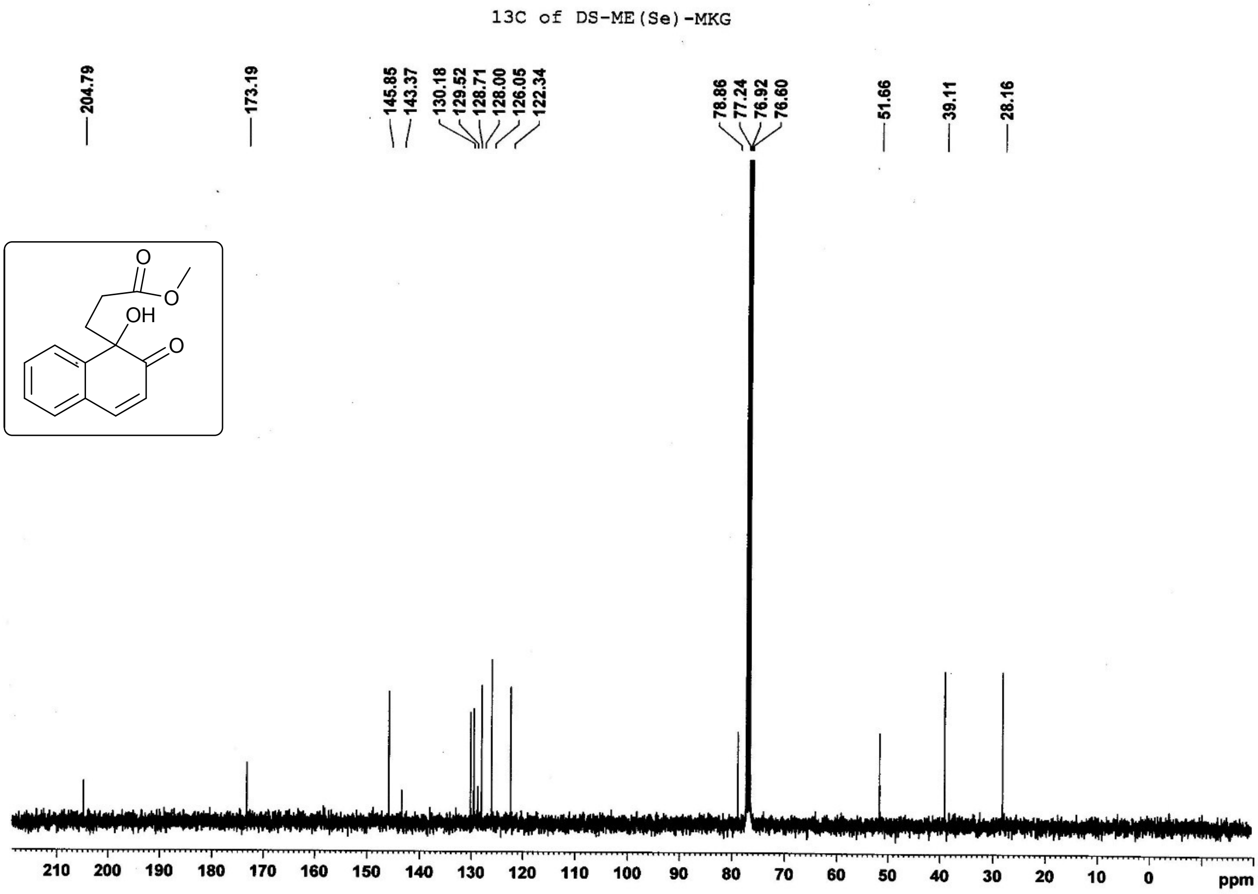
^{1}H NMR (400 MHz, CDCl$_3$) of compound 1(h)



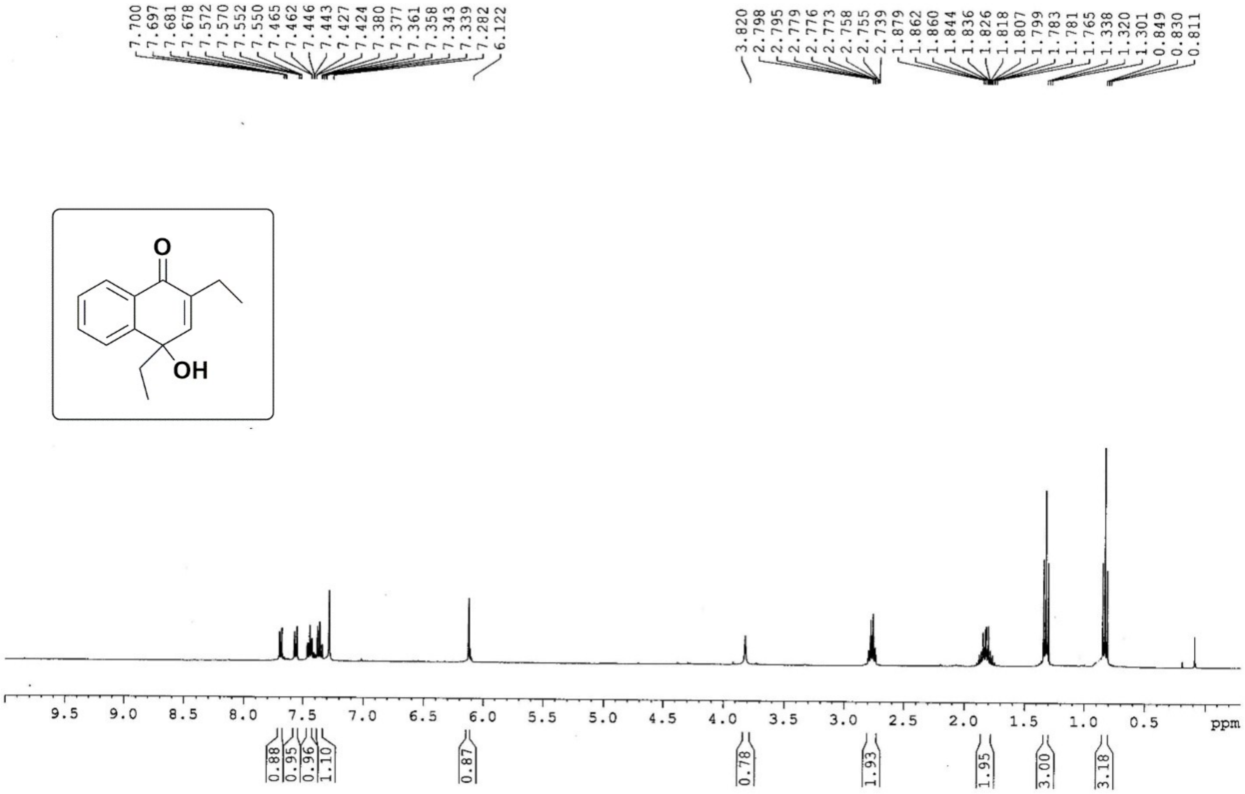
^{13}C NMR (100 MHz,$CDCl_3$) of compound 1(h)



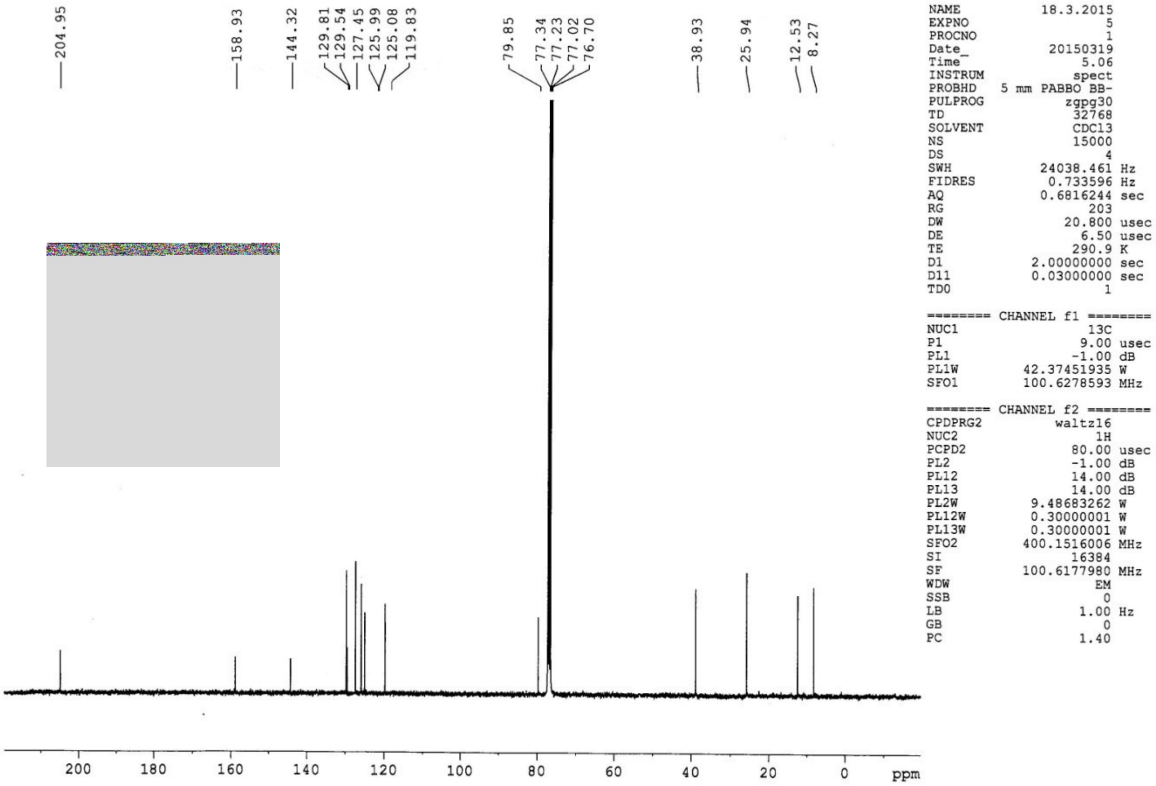
^{1}H NMR (400 MHz, $CDCl_3$) of compound 1(i)



^{13}C NMR (100 MHz,CDCl$_3$) of compound 1(i)



^{1}H NMR (400 MHz, CDCl$_3$) of 1(k)



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TD                 32768
SOLVENT            CDCl3
NS                 15000
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816244 sec
RG                   203
DW                20.800 usec
DE                  6.50 usec
TE                 290.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
PL1W         42.37451935 W
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.00 dB
PL13               14.00 dB
PL2W          9.48683262 W
PL12W         0.30000001 W
PL13W         0.30000001 W
SFO2         400.1516006 MHz
SI                 16384
SF           100.6177980 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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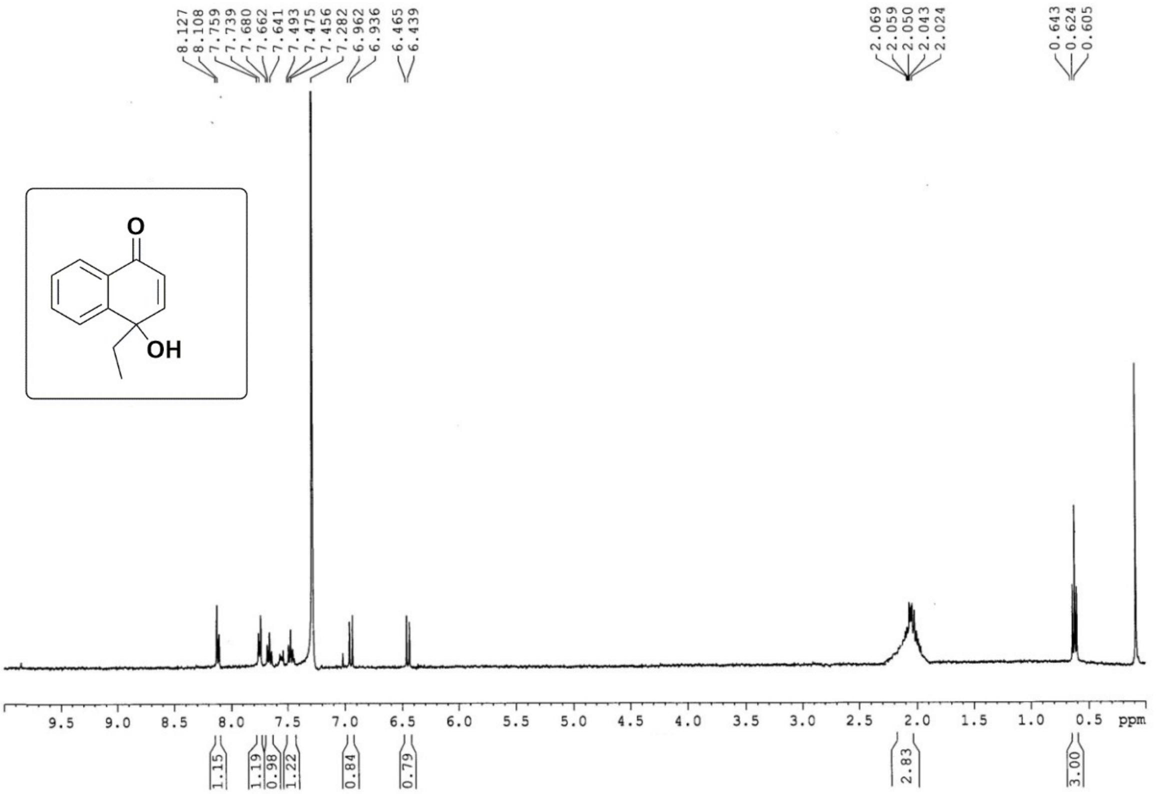
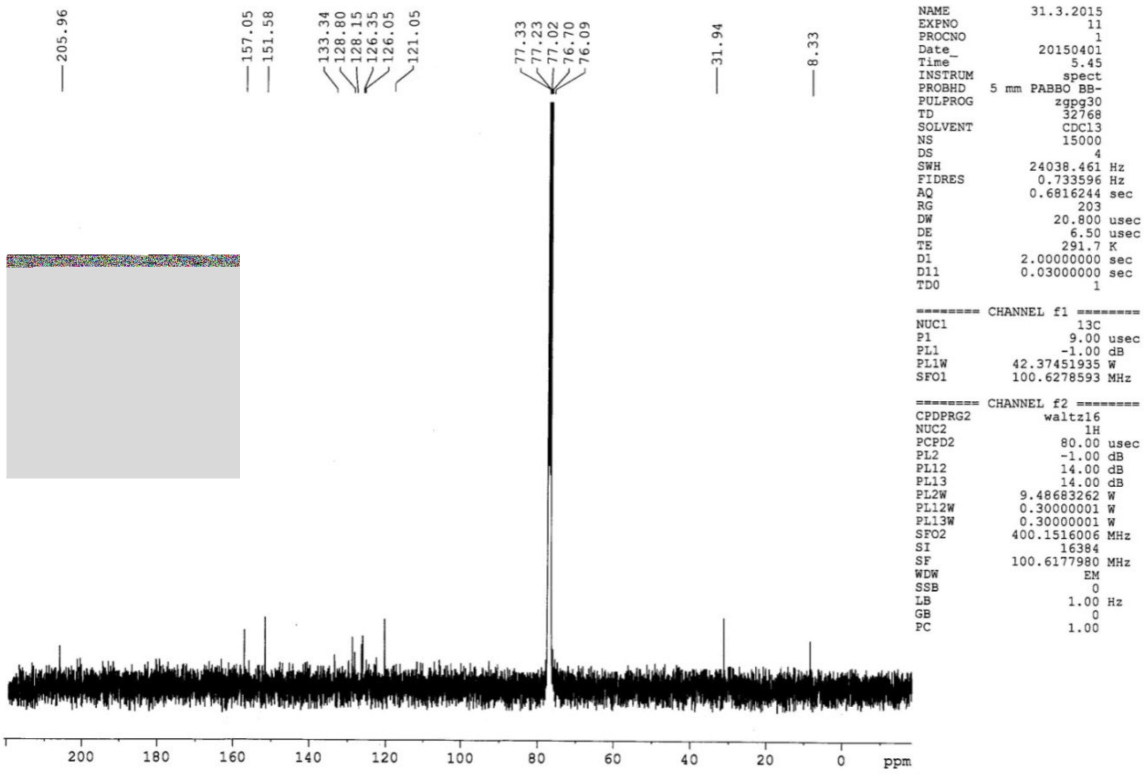
^{13}C NMR (100 MHz,CDCl$_3$) 1(k)



^{1}H NMR (400 MHz, $CDCl_3$) of 1(l)



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SOLVENT            CDCl3
NS                 15000
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
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RG                   203
DW                20.800 usec
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TE                 291.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
PL1W         42.37451935 W
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
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NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.00 dB
PL13               14.00 dB
PL2W          9.48683262 W
PL12W         0.30000001 W
PL13W         0.30000001 W
SFO2         400.1516006 MHz
SI                 16384
SF           100.6177980 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
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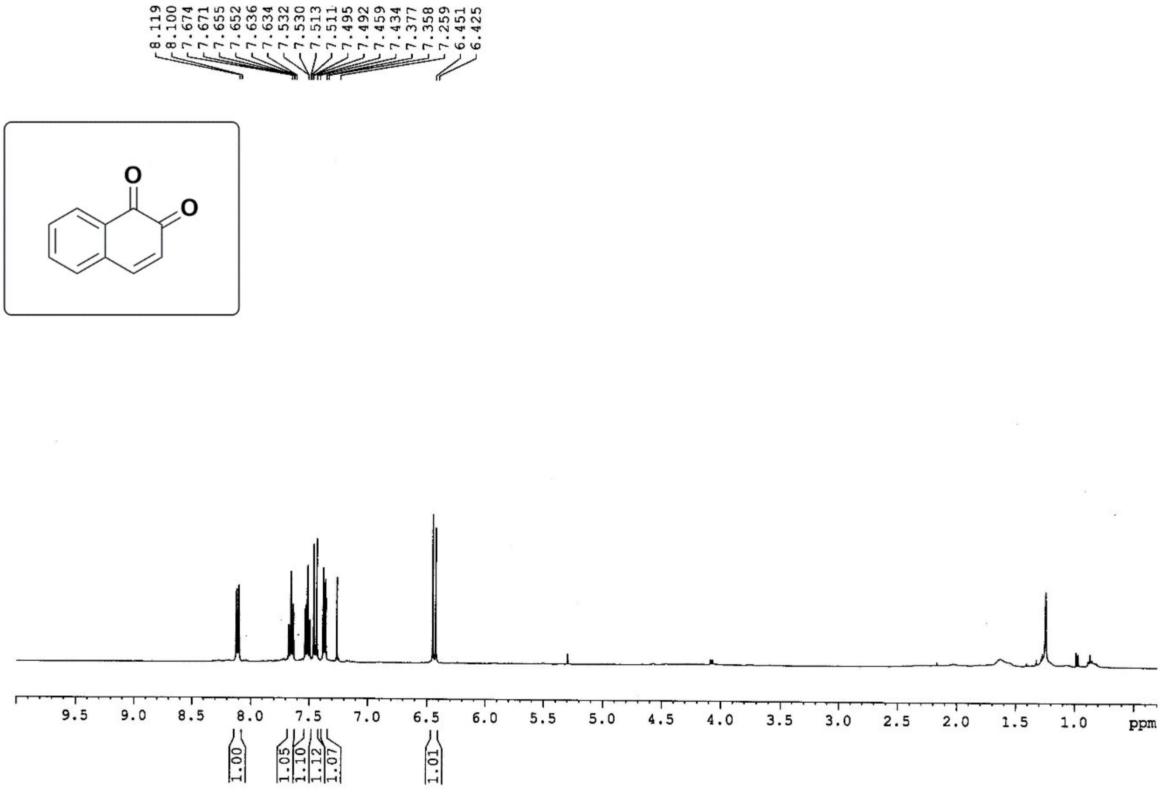
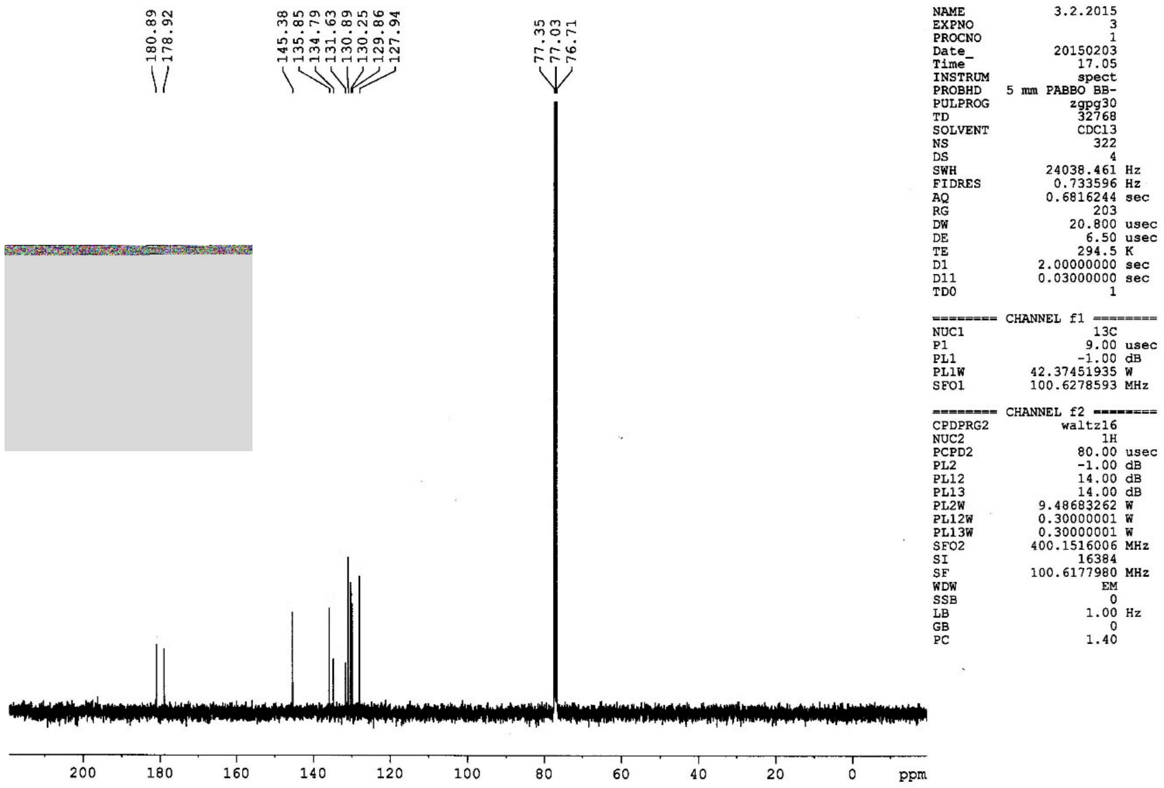
^{13}C NMR (100 MHz,CDCl$_3$) of 1(l)



^{1}H NMR (400 MHz, $CDCl_3$) of 1(j)



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TD                32768
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RG                  203
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TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PL1               -1.00 dB
PL1W        42.37451935 W
SFO1        100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              14.00 dB
PL13              14.00 dB
PL2W         9.48683262 W
PL12W        0.30000001 W
PL13W        0.30000001 W
SFO2        400.1516006 MHz
SI                16384
SF          100.6177980 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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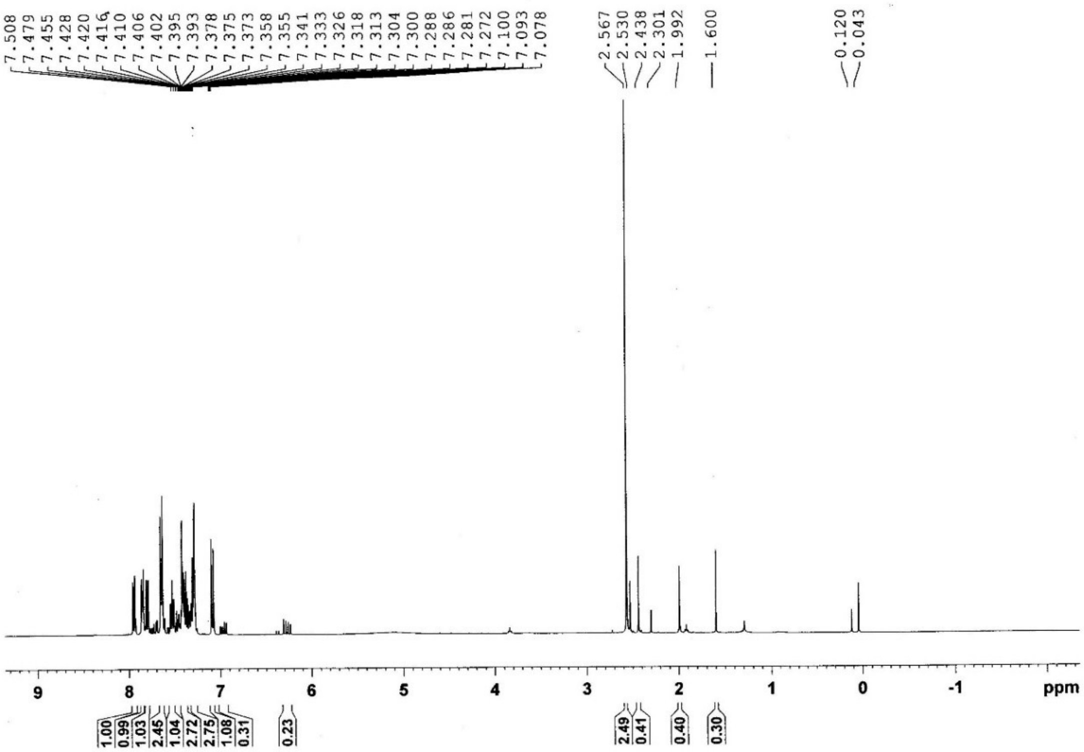
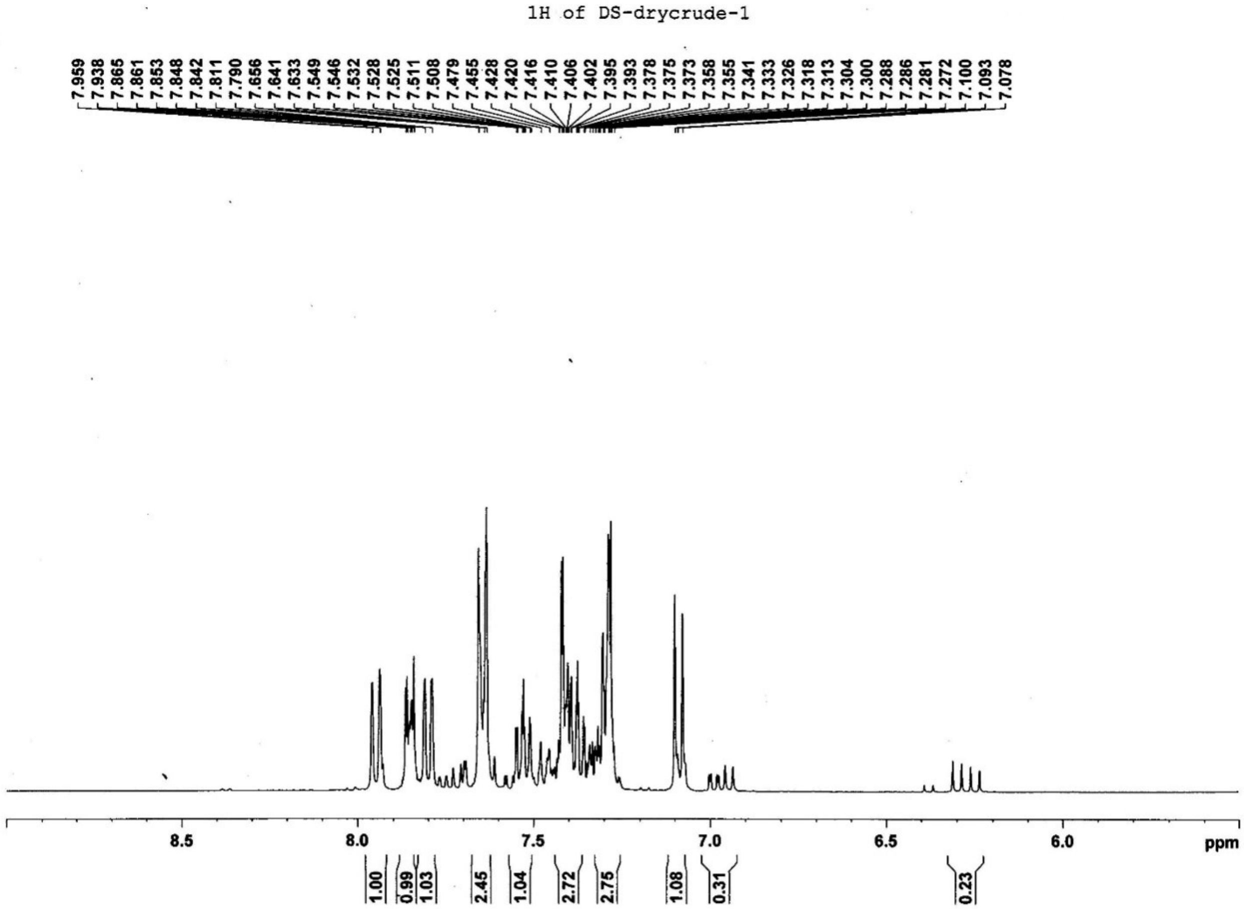
^{13}C NMR (100 MHz, CDCl$_3$) of 1(j)

Experimental spectra of crude reaction mixture (1-methyl-2-napthol as the starting compound)



^{1}H NMR of dry crude reaction mixture after 20 minute from reaction time


1H of DS-drycrude-1
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7.861
7.853
7.848
7.842
7.811
7.790
7.656
7.641
7.633
7.549
7.546
7.532
7.528
7.525
7.511
7.508
7.479
7.455
7.428
7.420
7.416
7.410
7.406
7.402
7.395
7.393
7.378
7.375
7.373
7.358
7.355
7.341
7.333
7.326
7.318
7.313
7.304
7.300
7.288
7.286
7.281
7.272
7.100
7.093
7.078
8.5
8.0
7.5
7.0
6.5
6.0
ppm
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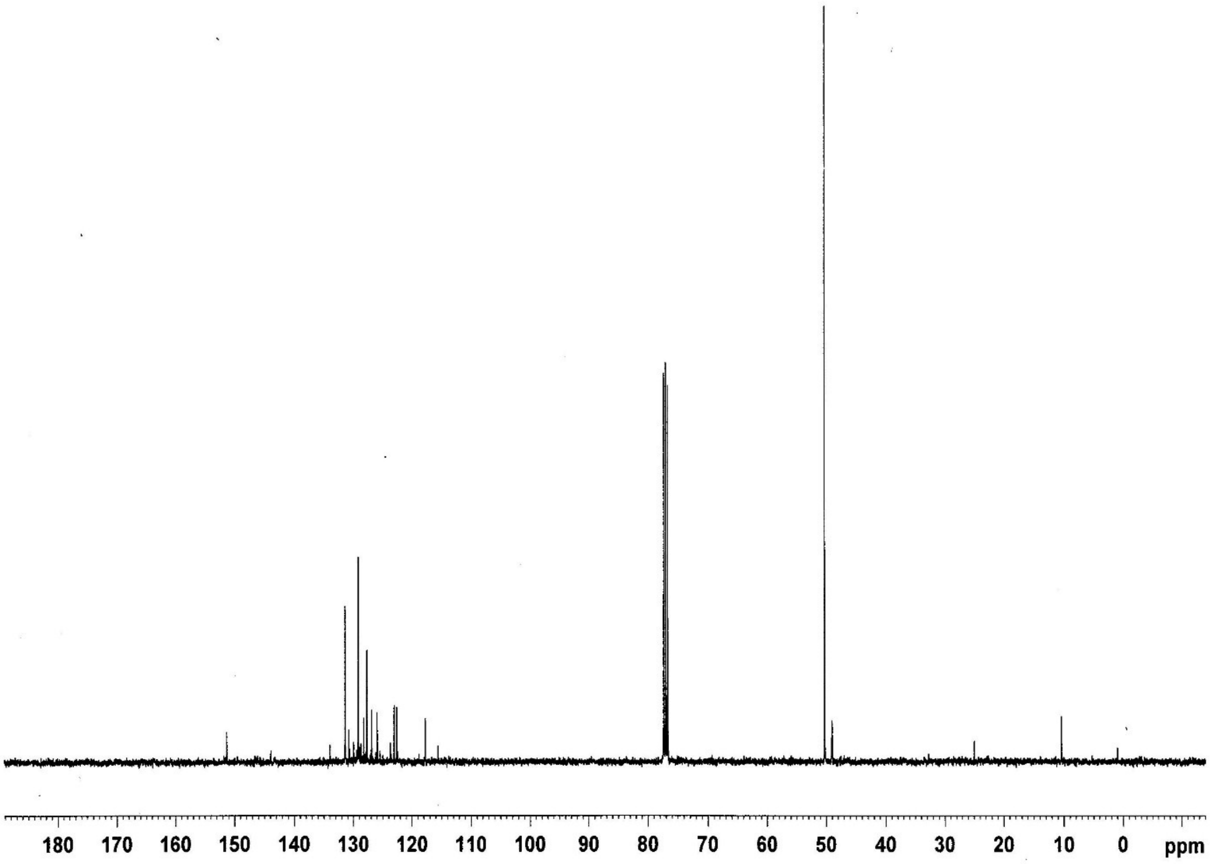
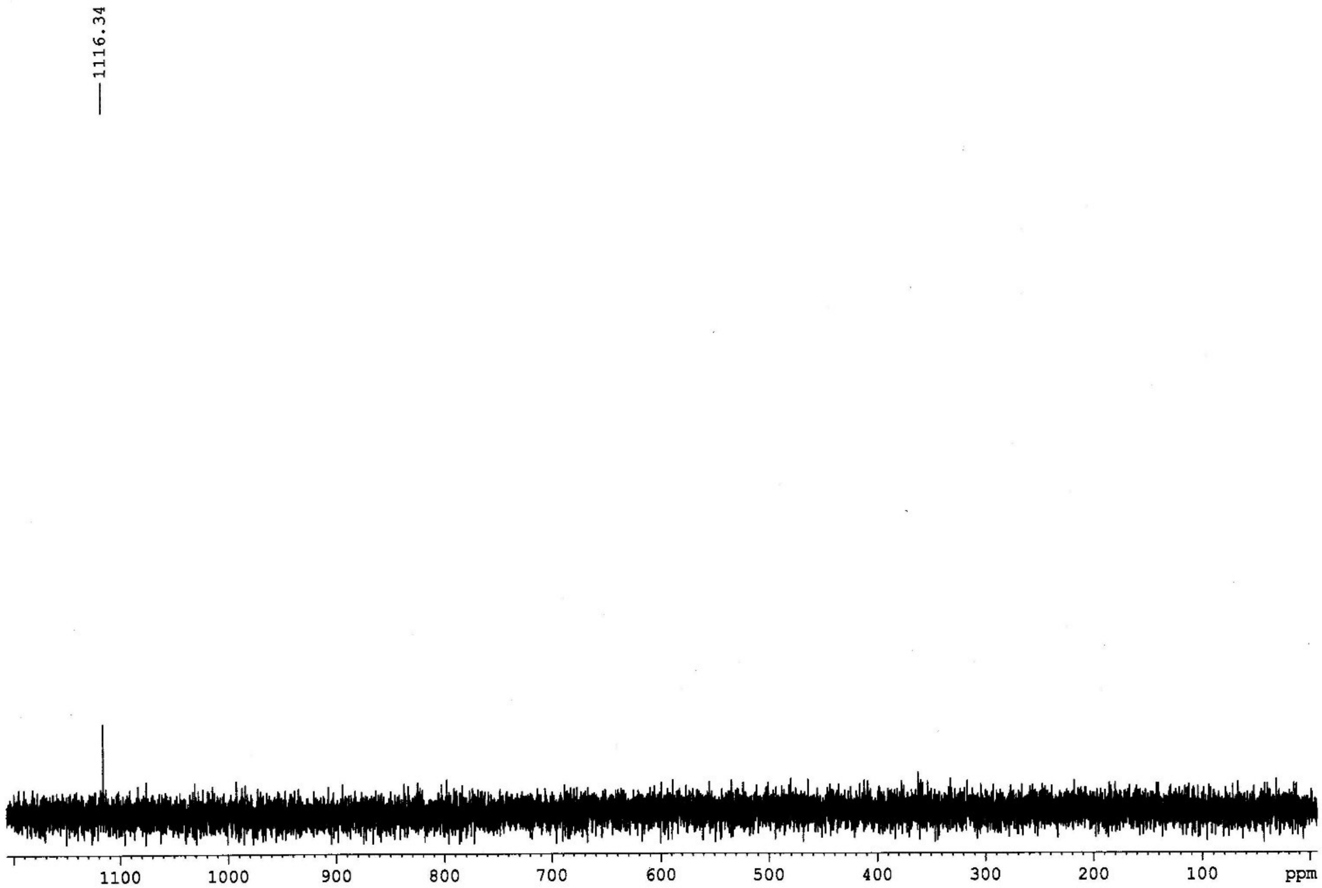
0.99
1.03
2.45
1.04
2.72
2.75
1.08
0.31
0.23

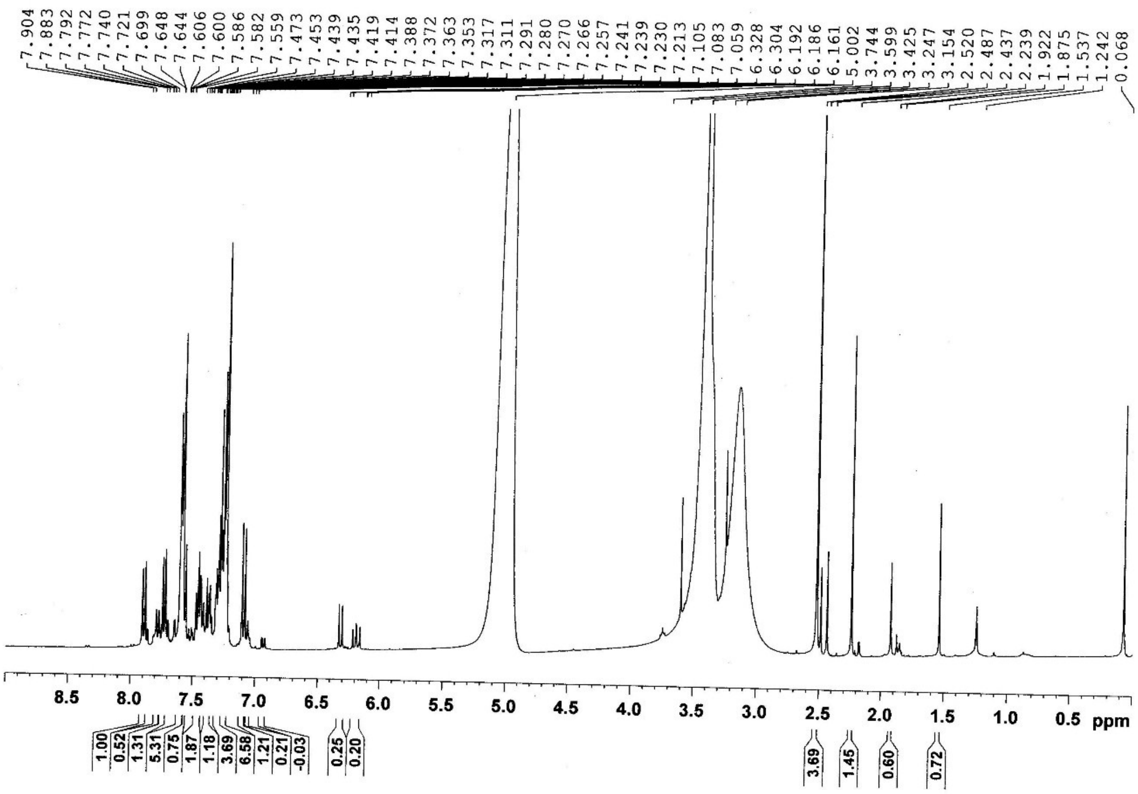
Expand view



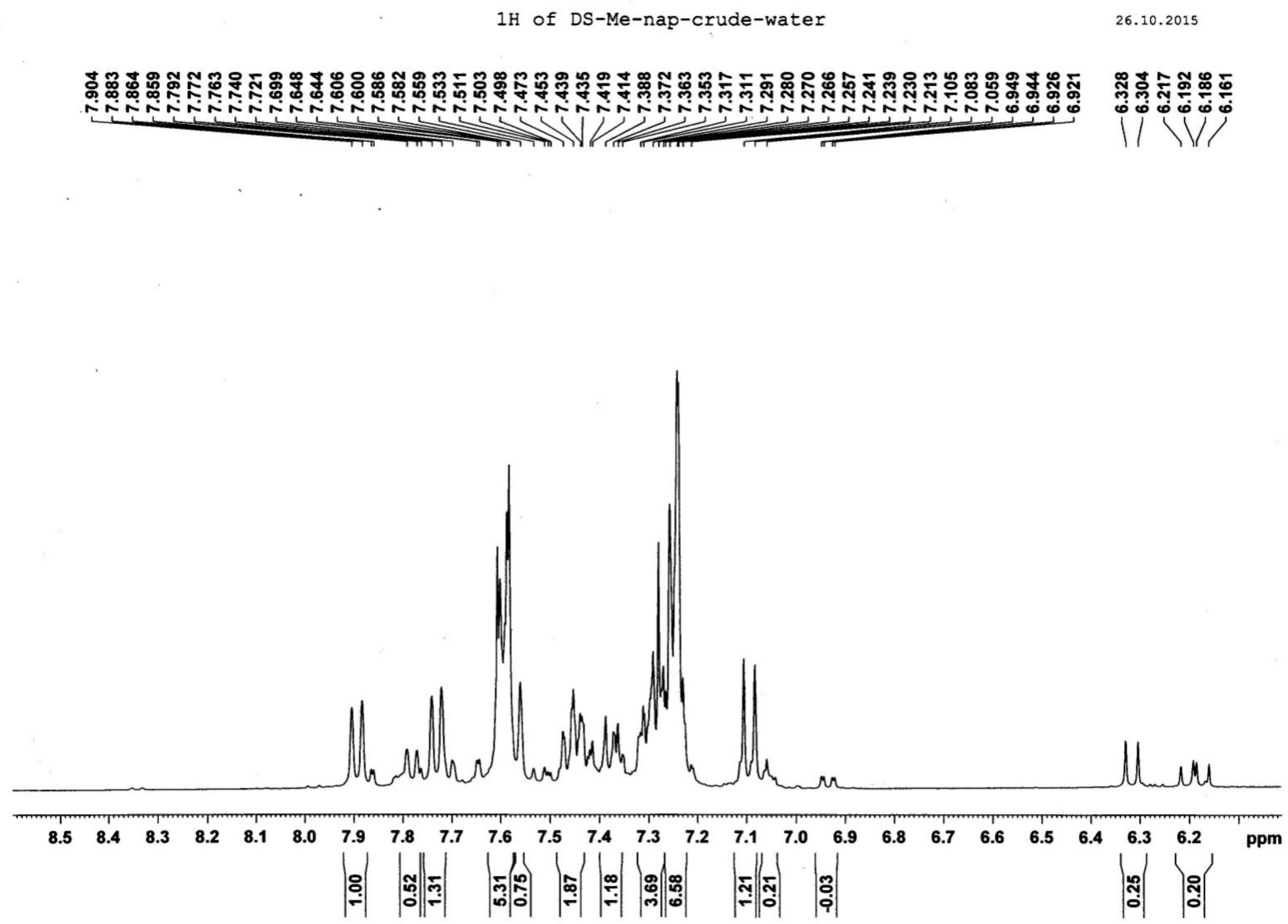
^{13}C NMR of dry crude reaction mixture after 20 minute from reaction time



Selenium (^{77}Se) NMR of reaction mixture before addition of water



^{1}H NMR of crude reaction mixture with addition of 1-drop water & methanol after 30 minute from reaction time


1H of DS-Me-nap-crude-water
26.10.2015
7.904
7.883
7.864
7.859
7.792
7.772
7.763
7.740
7.721
7.699
7.648
7.644
7.606
7.600
7.586
7.582
7.559
7.533
7.511
7.503
7.498
7.473
7.453
7.439
7.435
7.419
7.414
7.388
7.372
7.363
7.353
7.317
7.311
7.291
7.280
7.270
7.266
7.257
7.241
7.239
7.230
7.213
7.105
7.083
7.059
6.949
6.944
6.926
6.921
6.328
6.304
6.217
6.192
6.186
6.161
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
6.4
6.3
6.2
ppm
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1.31
5.31
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1.18
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6.58
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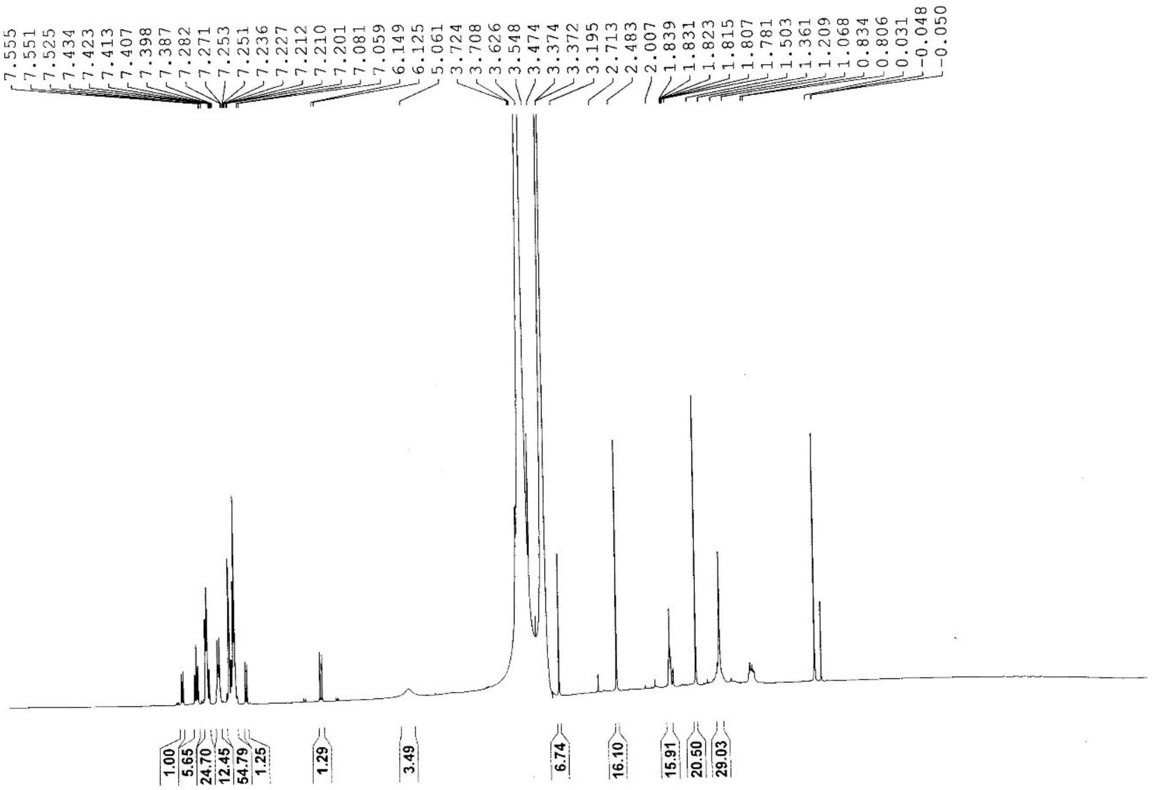
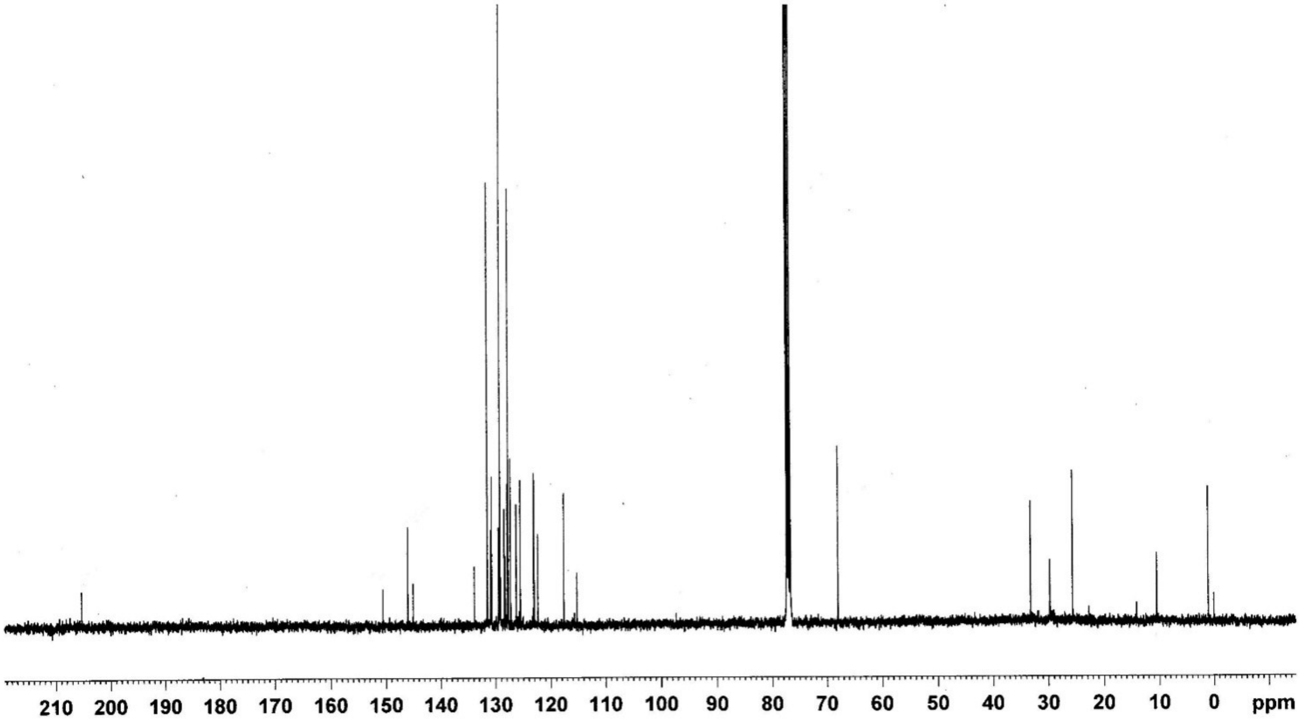
0.25
0.20

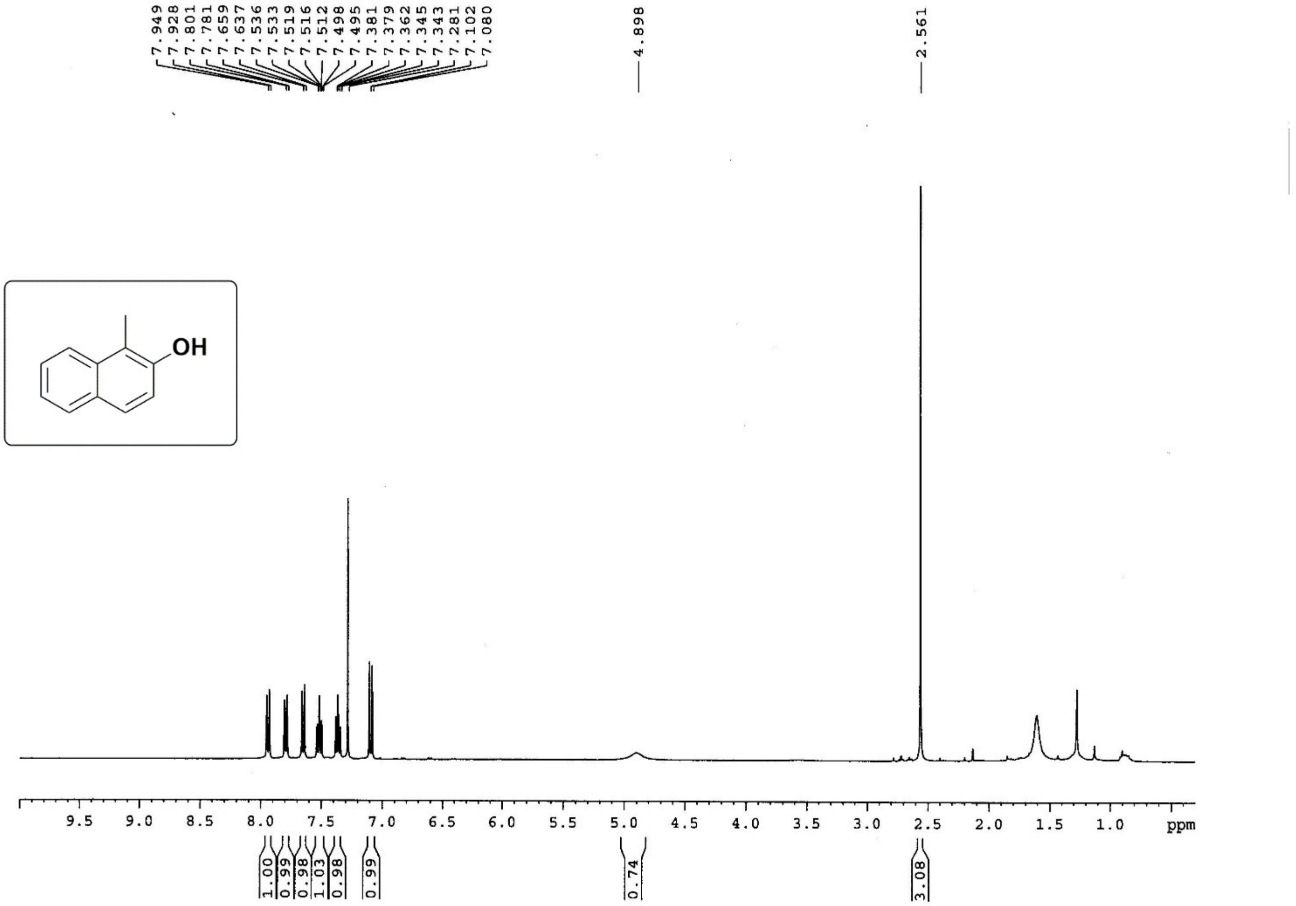
Expand view



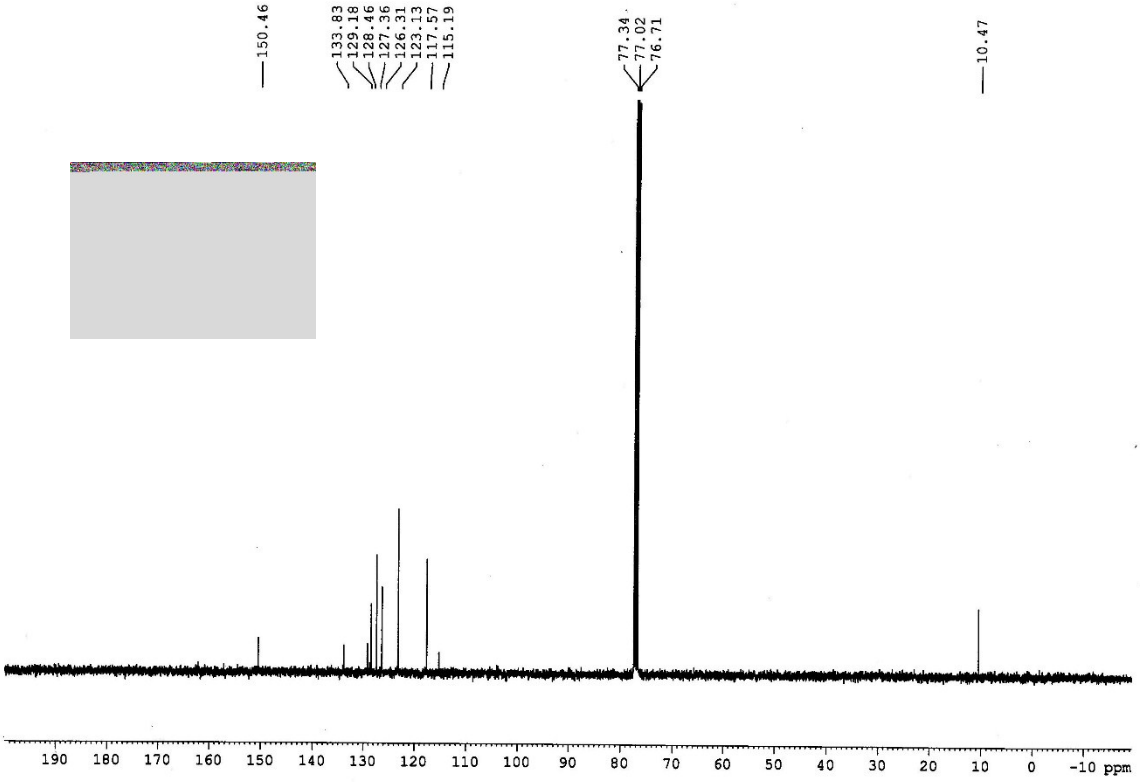
^{1}H NMR of crude reaction mixture after 30 minute from the addition of water in the reaction mixture



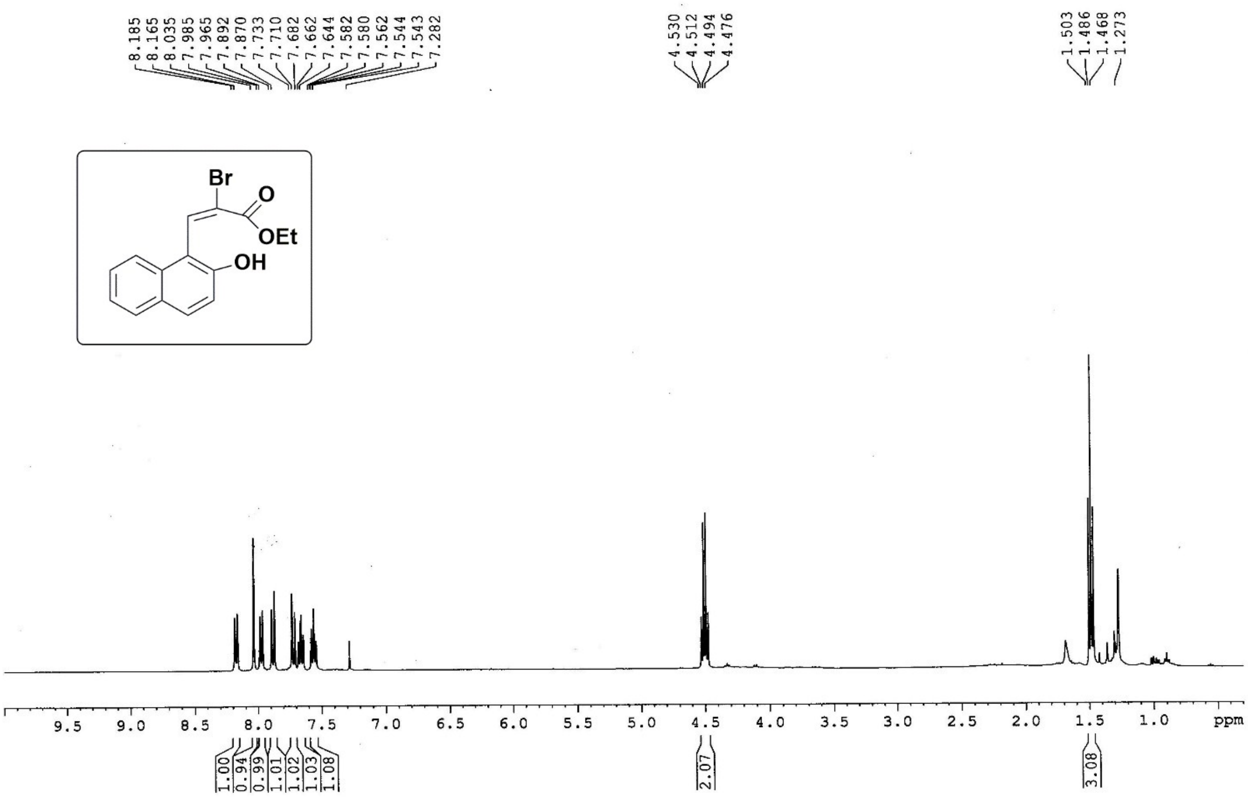
^{13}C NMR of crude reaction mixture after 30 minute from the addition of water in the reaction mixture



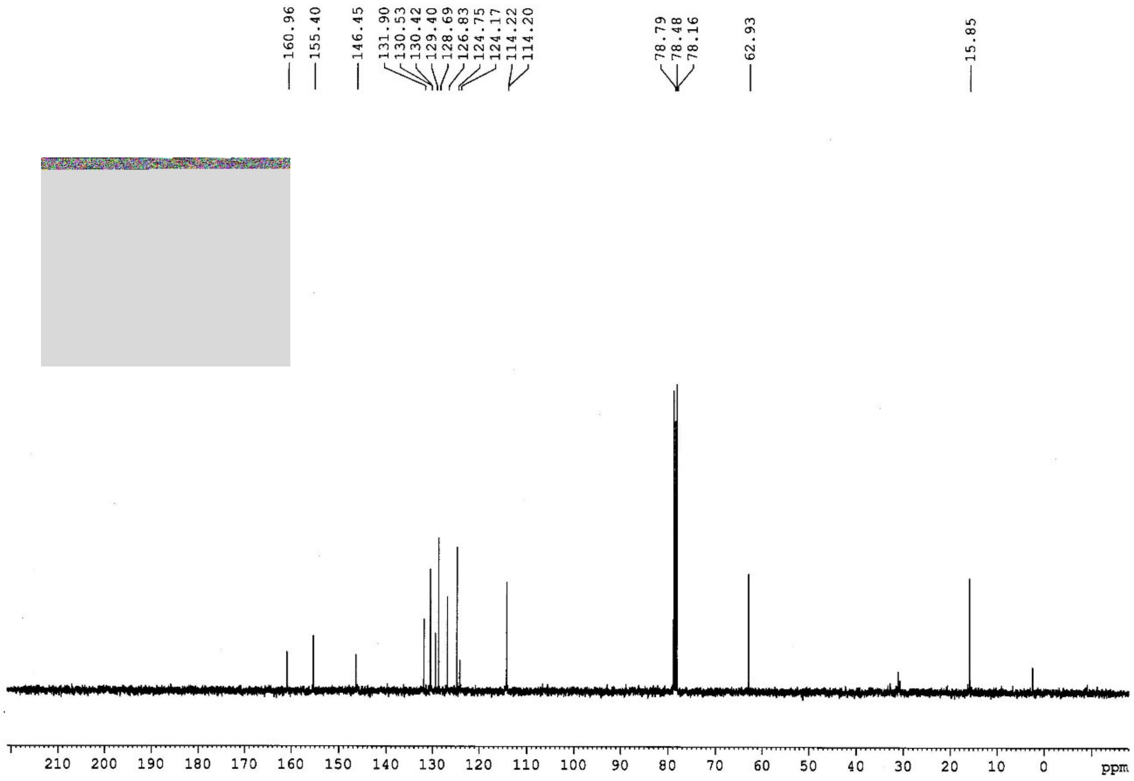
^{1}H NMR (400 MHz, $CDCl_3$) of starting 1-methyl-2-naphthol



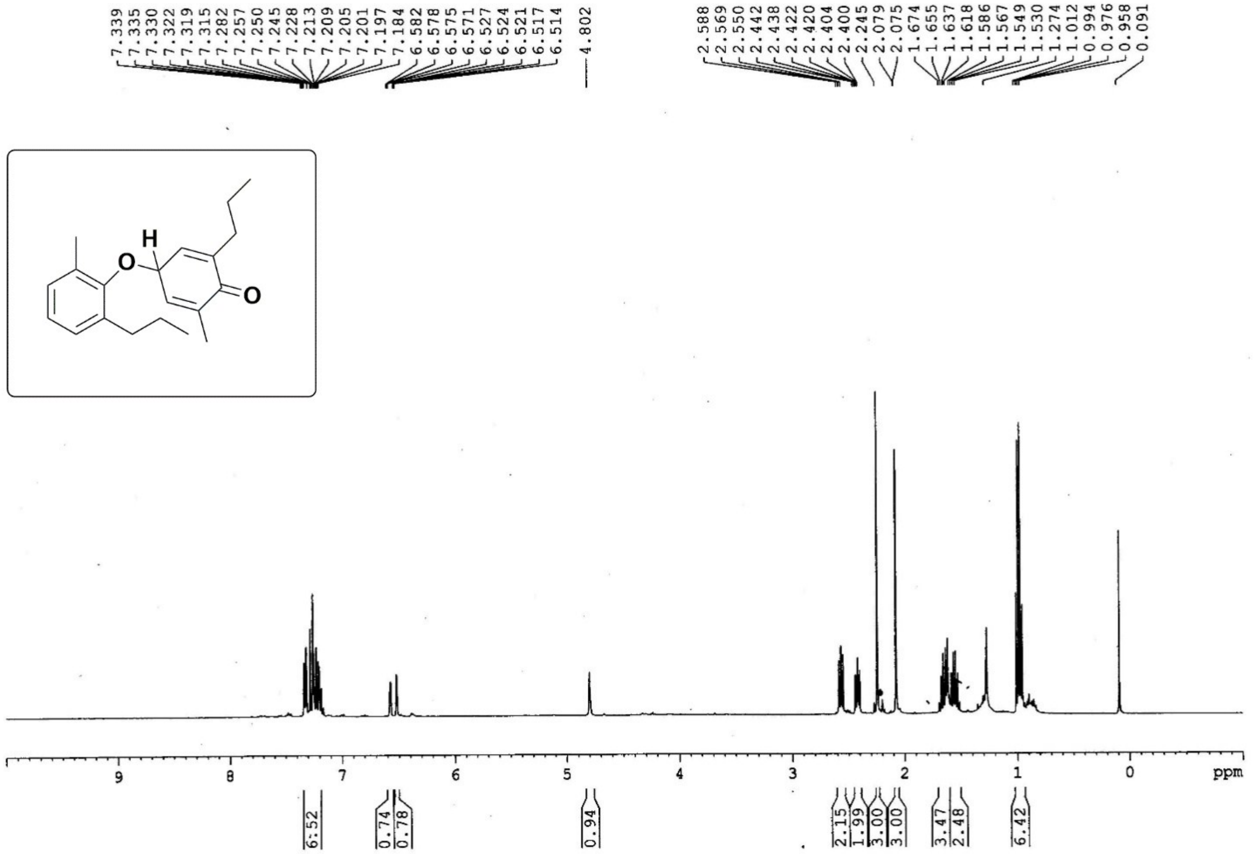
^{13}C NMR (100 MHz, $CDCl_3$) of starting 1-methyl-2-naphthol



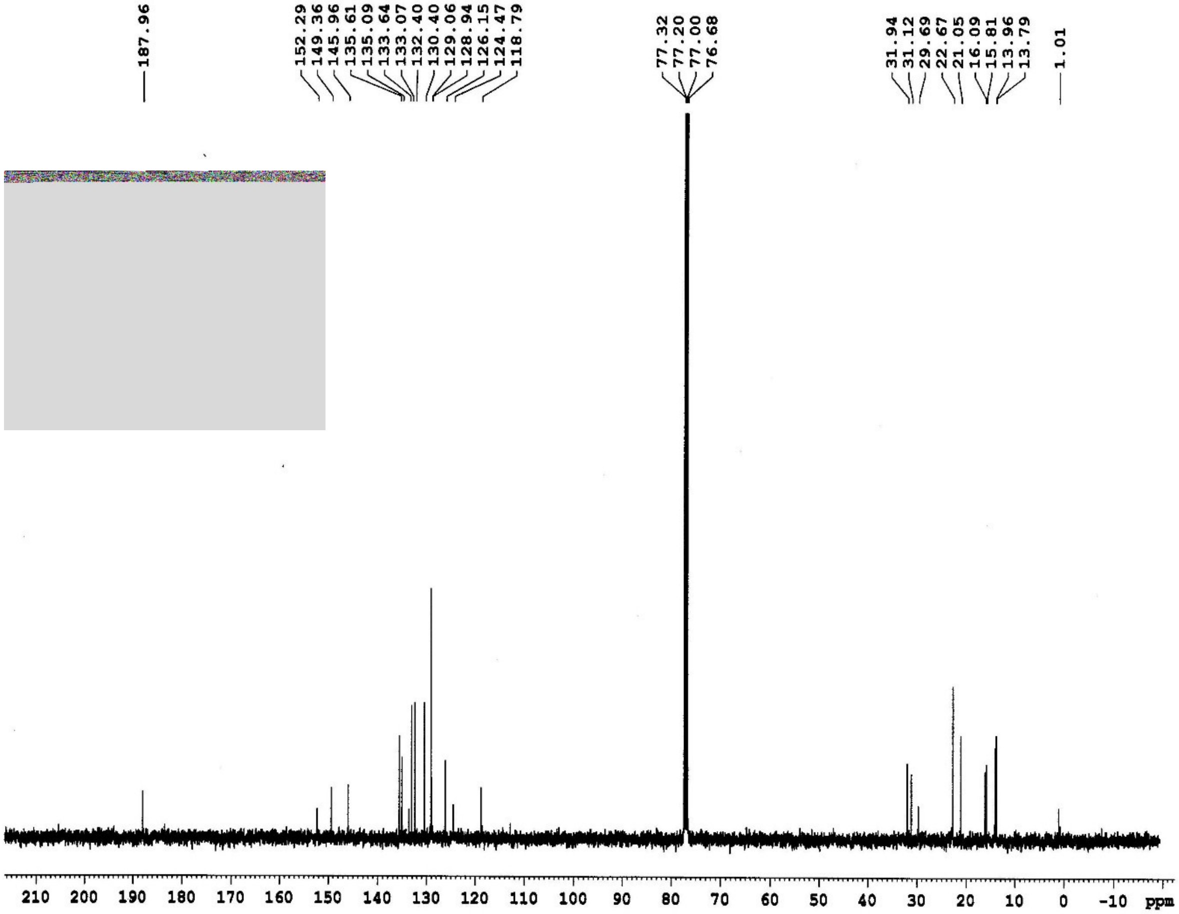
^{1}H NMR (400 MHz, $CDCl_3$) of 1(m)



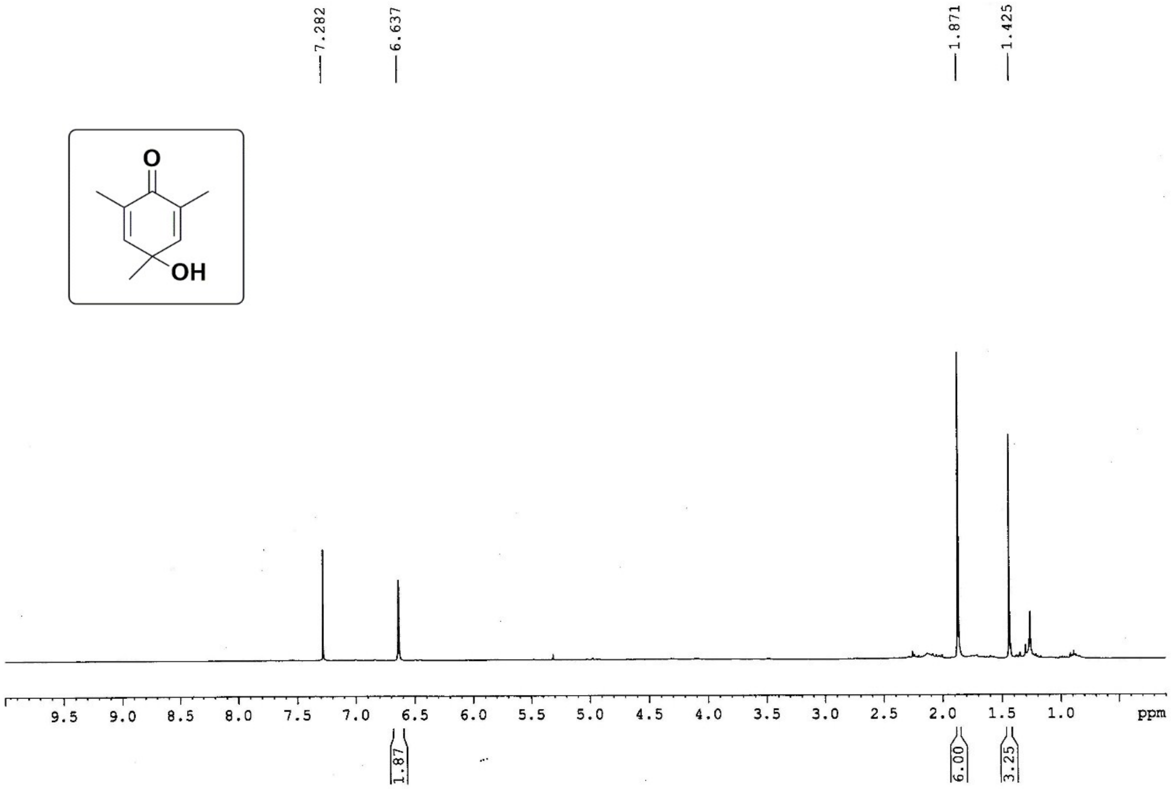
^{13}C NMR (100 MHz,CDCl$_3$) of 1(m)



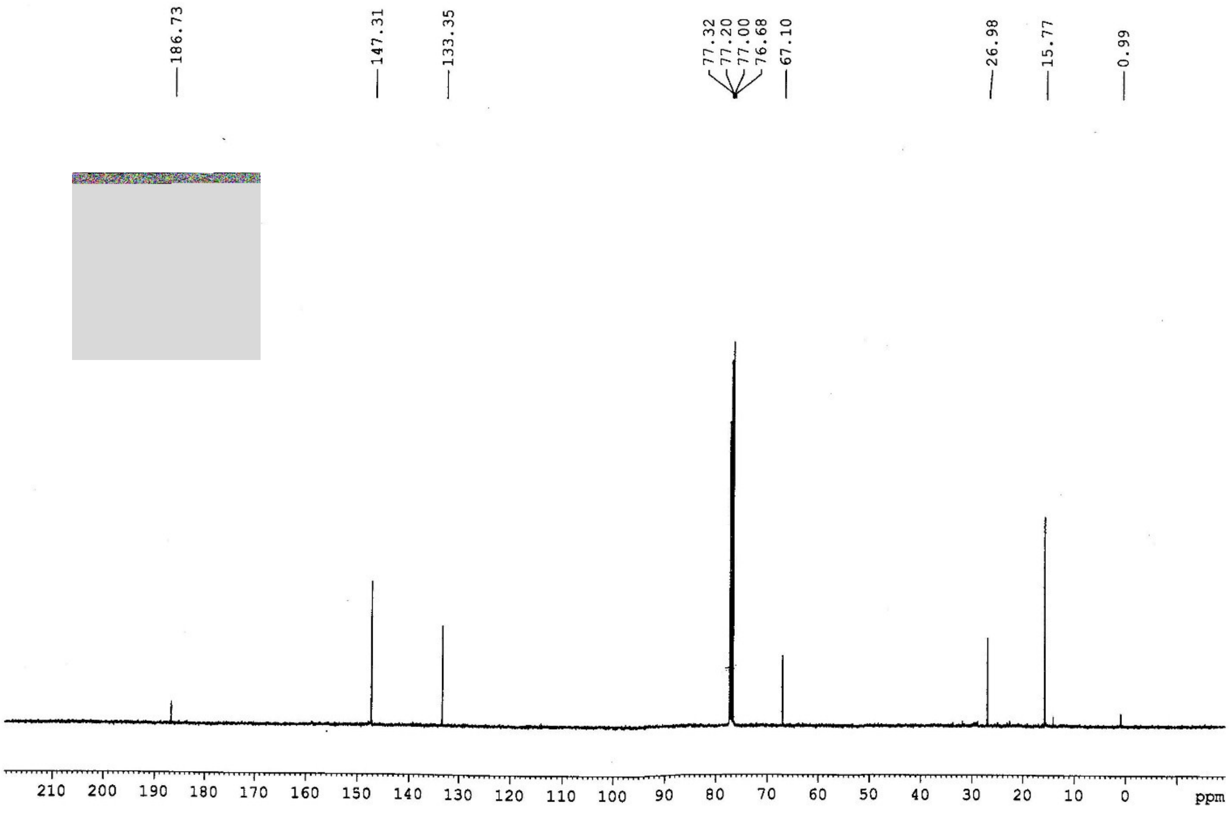
^{1}H NMR (400 MHz, $CDCl_3$) of 2(a)



^{13}C NMR (100 MHz, CDCl$_3$) of 2(a)

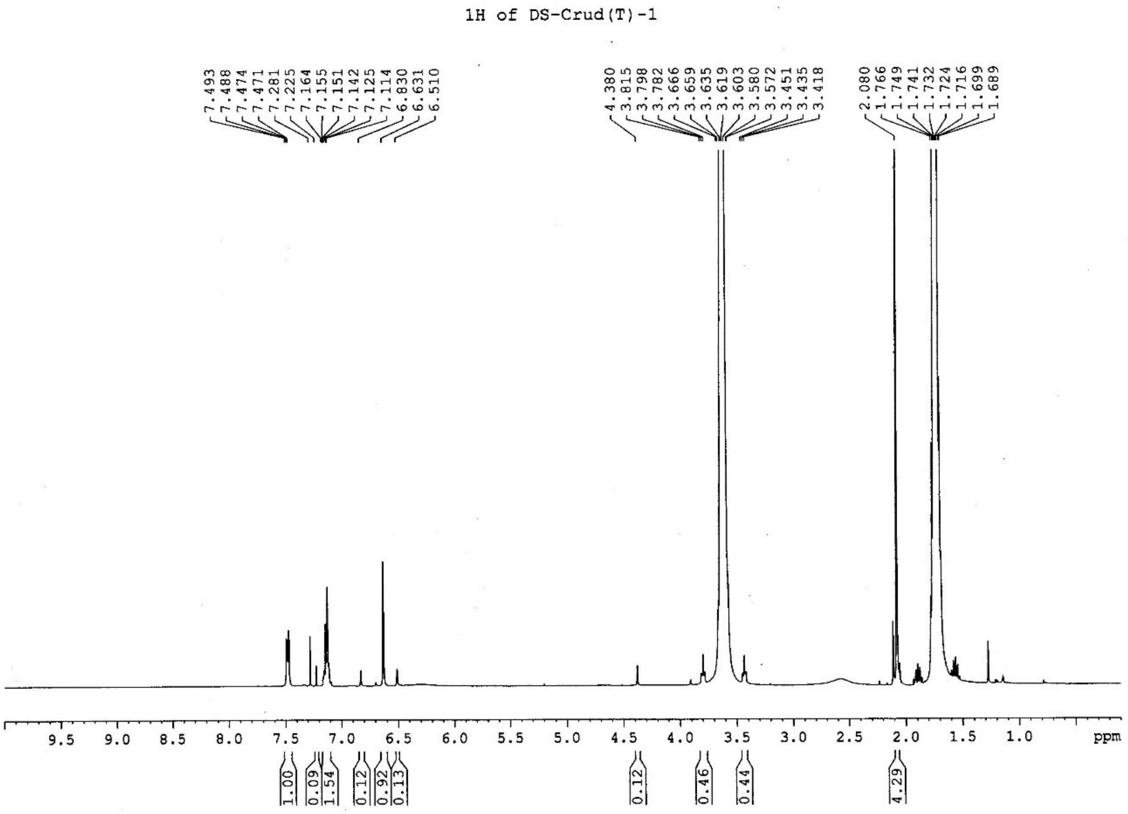


^{1}H NMR (400 MHz, CDCl$_3$) of 2(b)

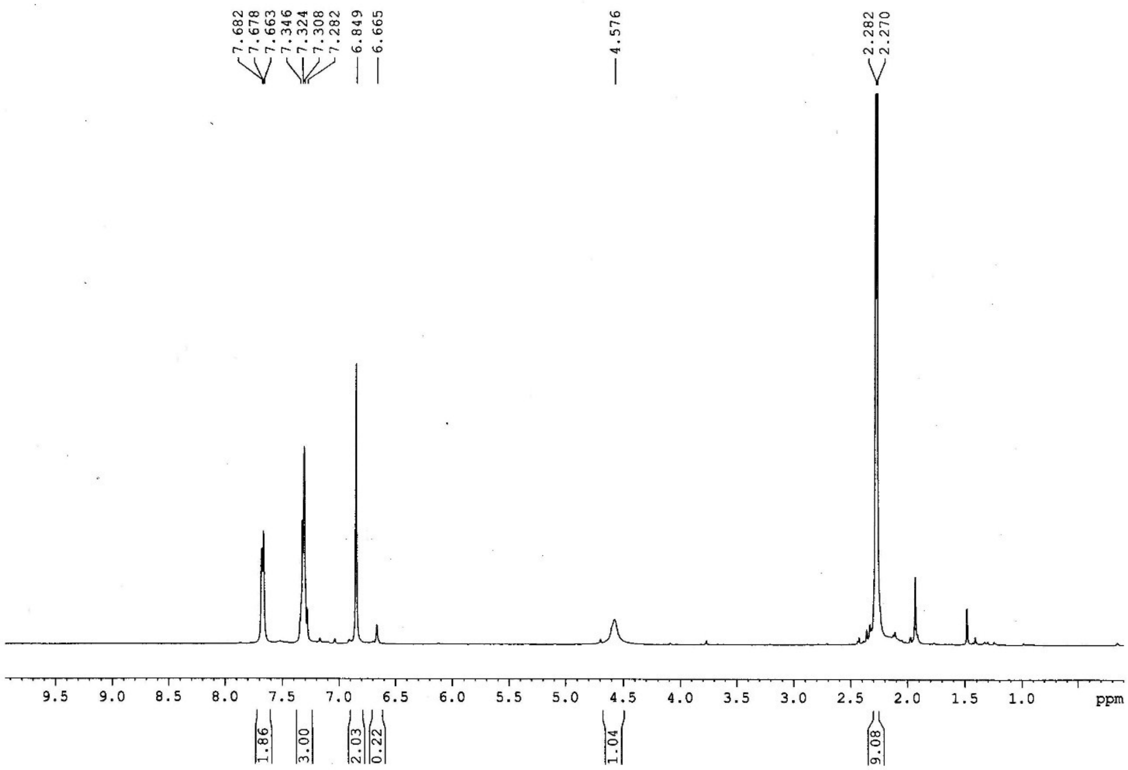


^{13}C NMR (100 MHz, CDCl$_3$) of 2(b)

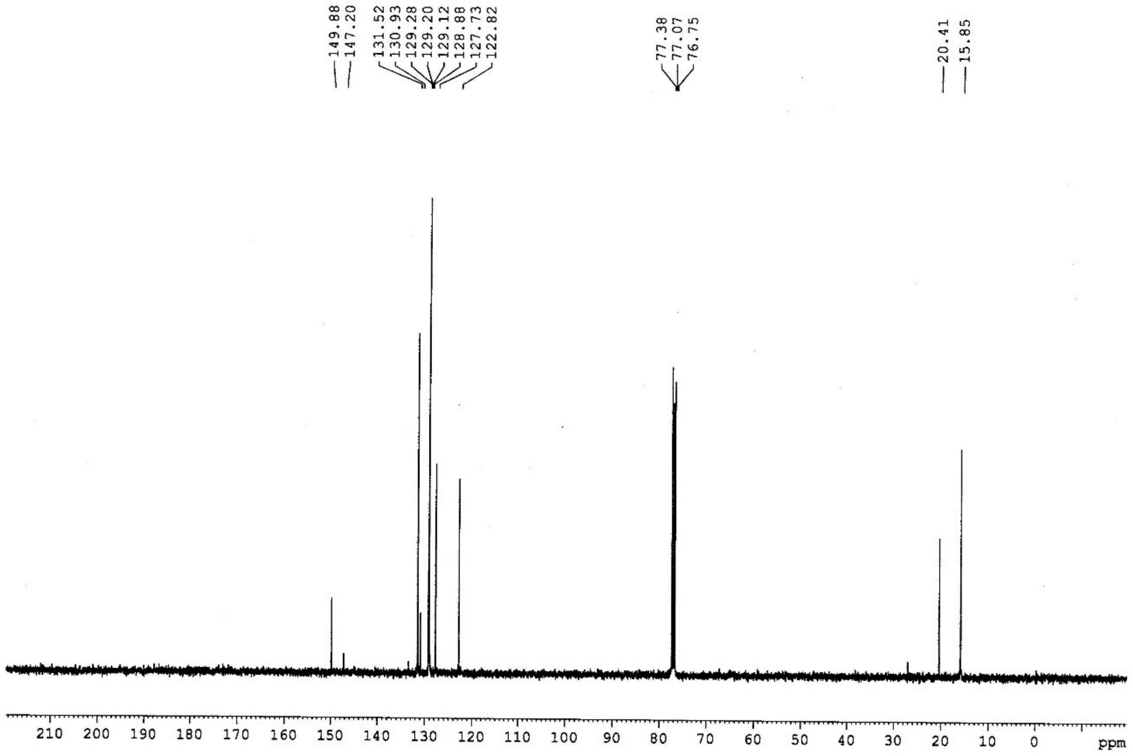
Insitu-experimental spectra of 2,4,6-trimethyl phenol



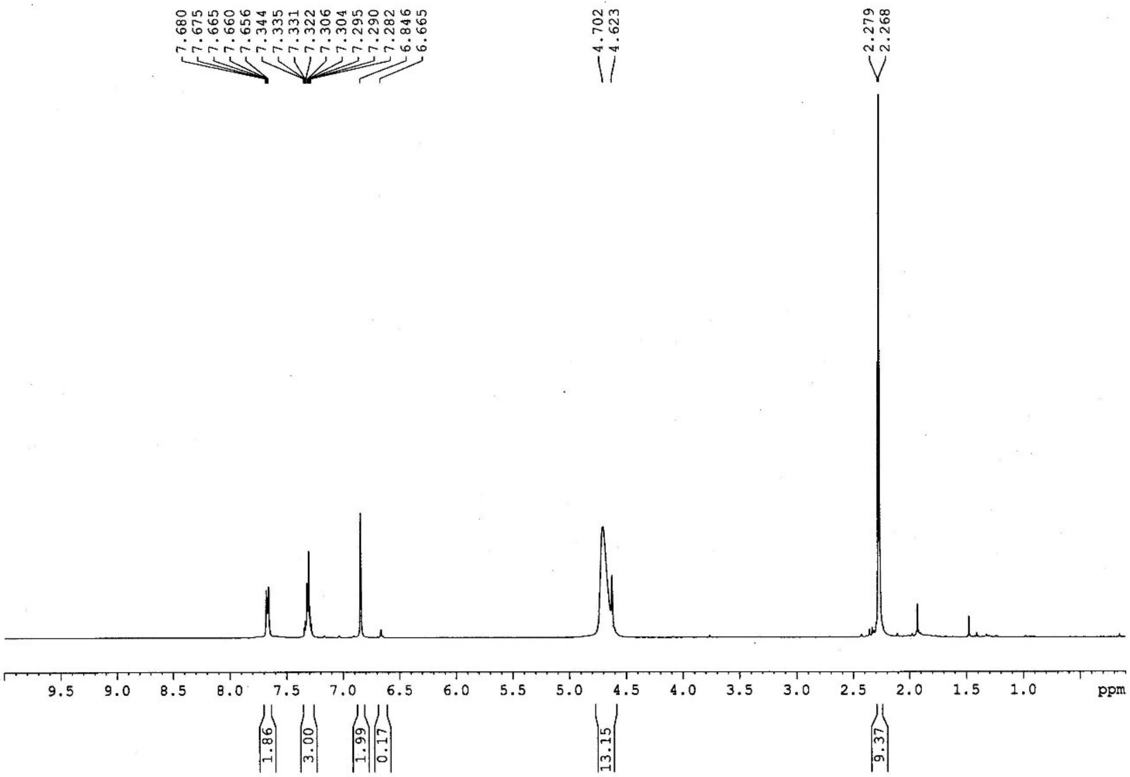
^{1}H NMR of crude reaction mixture (with THF solvent) of 2,4,6-trimethyl-phenol after 30 minute from reaction time



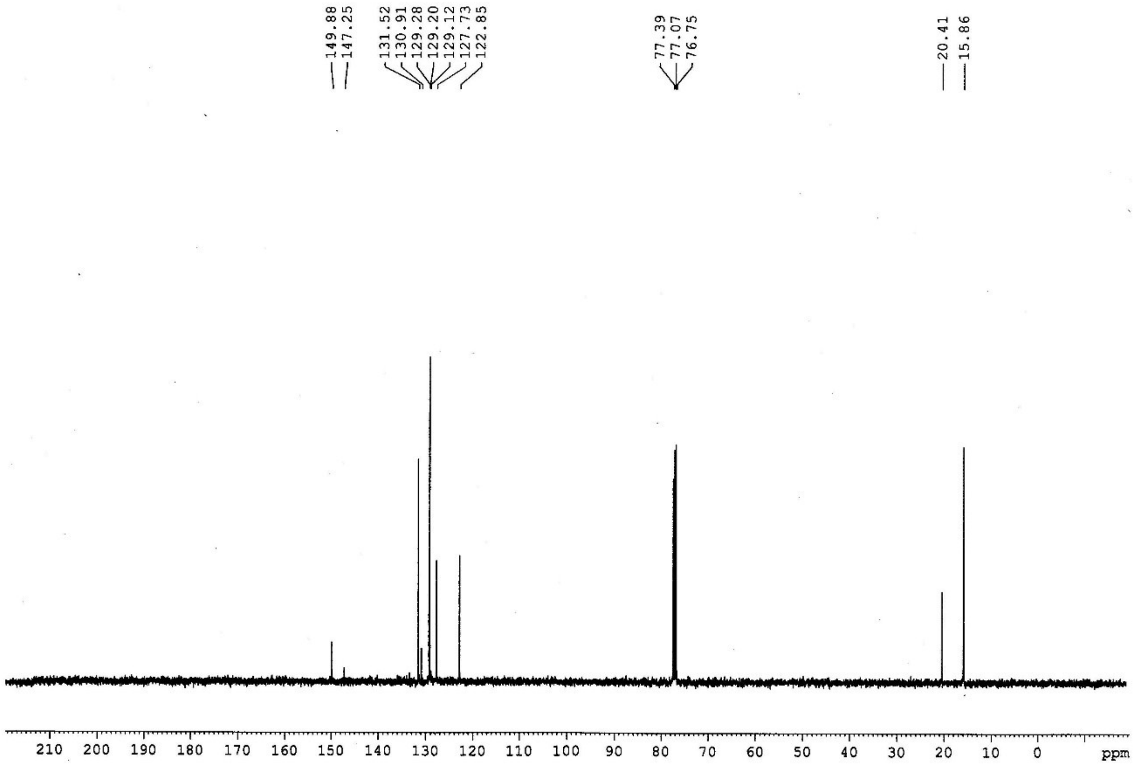
^1H NMR of crude reaction mixture (without solvent) of 2,4,6-trimethyl-phenol after 30 minute from reaction time



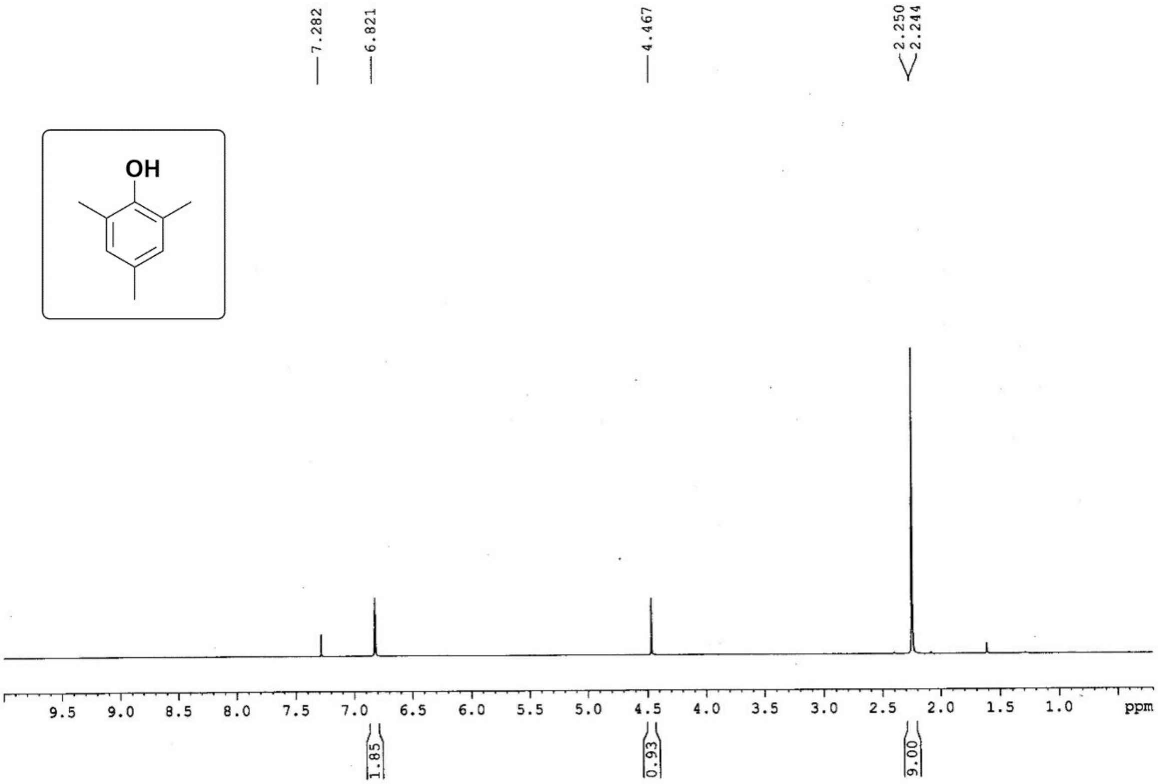
^{13}C NMR of crude reaction mixture (without solvent) of 2, 4, 6-trimethyl-phenol after 30 minute from reaction time.



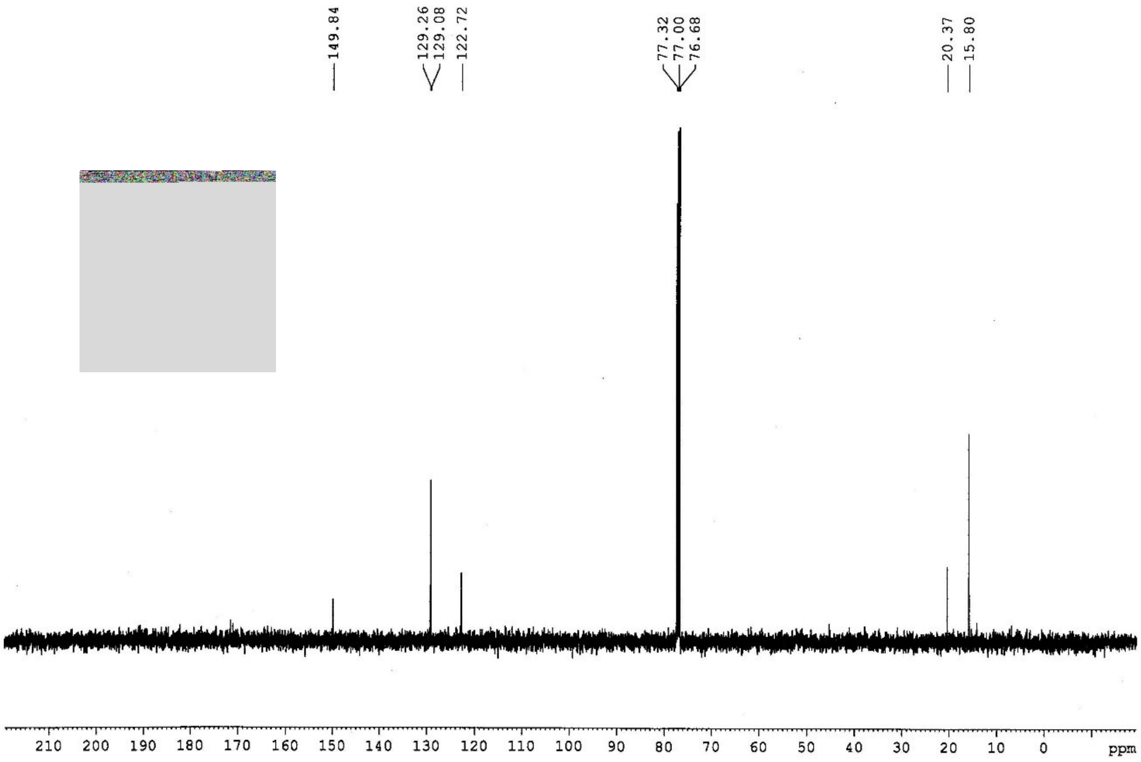
^{1}H NMR of crude reaction mixture of 2,4,6-trimethyl-phenol after addition of water in NMR tube



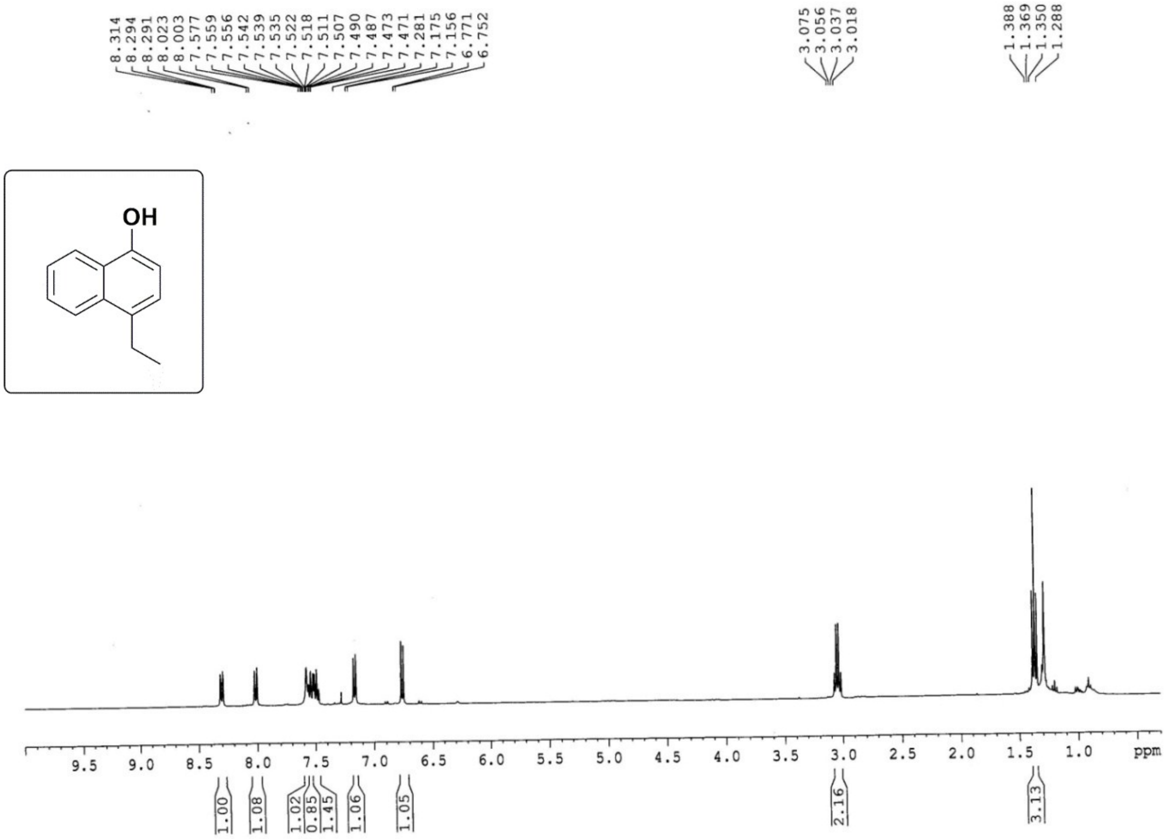
^{13}C NMR of crude reaction mixture of 2,4,6-trimethyl-phenol after addition of water in NMR tube



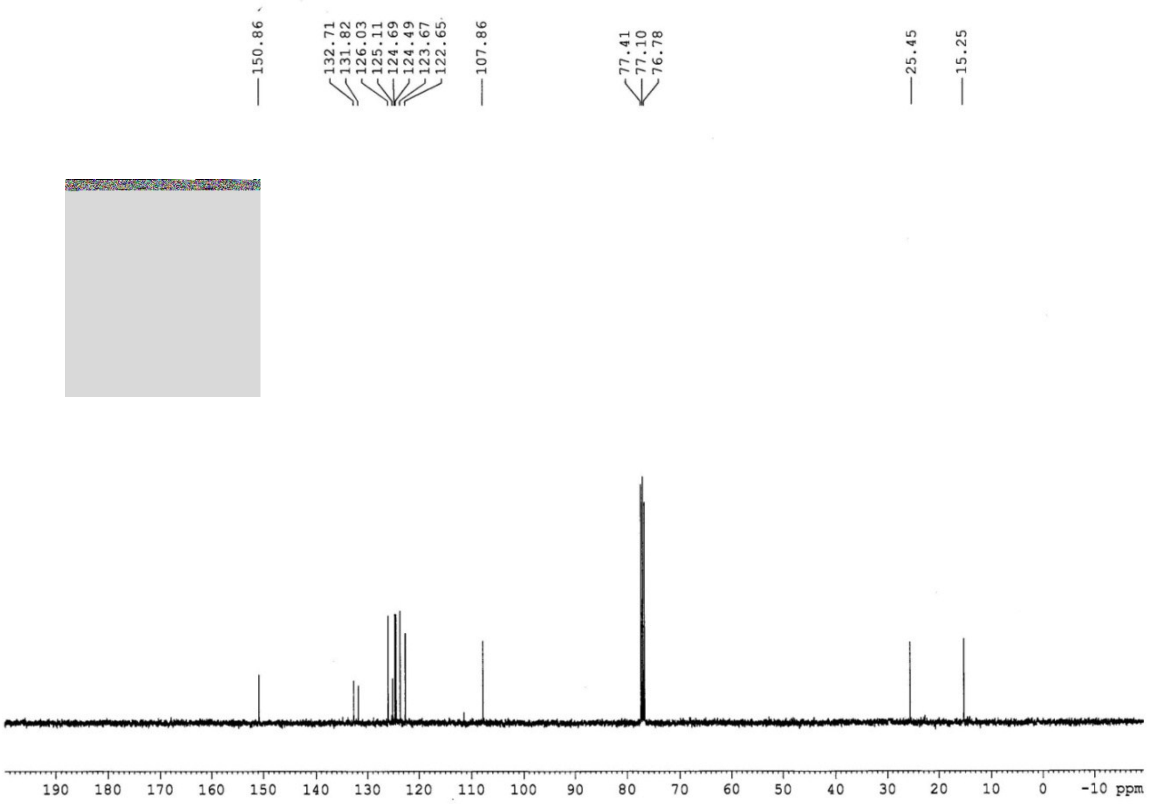
^{1}H NMR of 2,4,6-trimethyl phenol



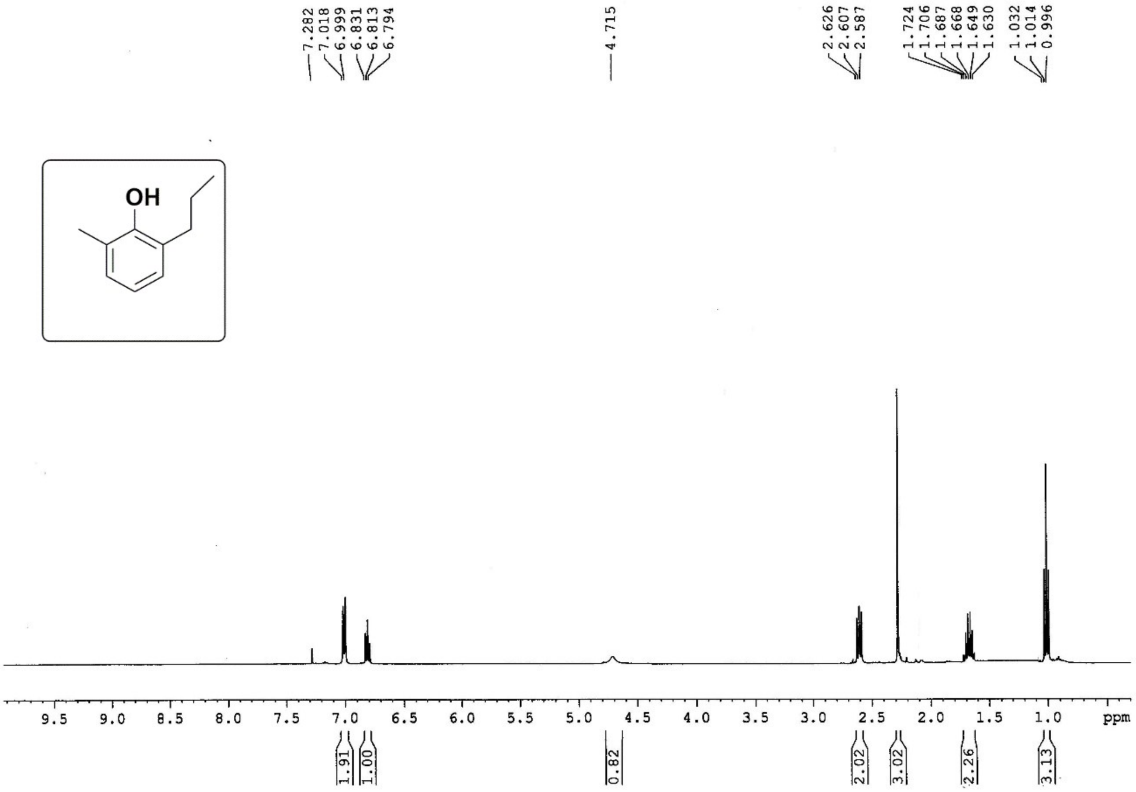
^{13}C NMR (100 MHz, CDCl$_3$) of 2,4,6-trimethyl phenol



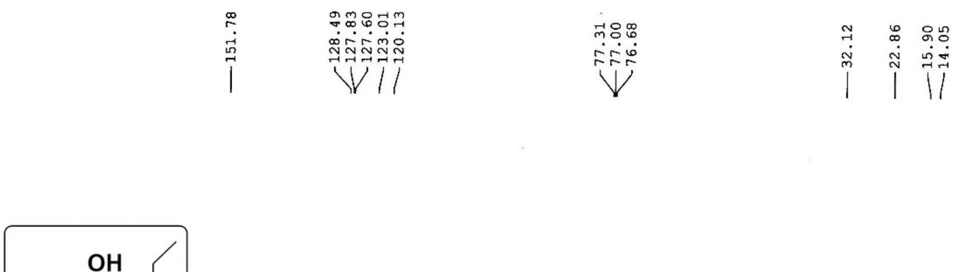
^{1}H NMR (400 MHz, $CDCl_3$) of 4-ethylnaphthalen-1-ol (s_{14})



^{13}**C NMR (100 MHz, CDCl$_3$) of 4-ethylnaphthalen-1-ol (s$_{14}$)**



^{1}H NMR (400 MHz, CDCl$_3$) of (S$_{16}$)



^{13}C NMR (100 MHz, CDCl$_3$) of (S_{16})